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Monsanto Research Corp.
Dayton, OH

Prepared for

Industrial Environmental Research Lab.
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PREFACE

The Industrial Environmental Research Laboratory (IERL) of the U.S. Environmental Protection Agency (EPA) has the responsibility for insuring that pollution control technology is available for stationary sources to meet the requirements of the Clean Air act, the Federal Water Pollution Control Act, and solid waste legislation. If control technology is unavailable, inadequate, or uneconomical, then financial support is provided for the development of the needed control techniques for industrial and extractive process industries. The Chemical Processes Branch of the Industrial Processes Division of IERL has the responsibility for investing tax dollars in programs to develop control technology for a large number of operations (more than 500) in the chemical industries.

Monsanto Research Corporation (MRC) has contracted with EPA to investigate the environmental impact of various industries which represent sources of pollution in accordance with EPA's responsibility as outlined above. Dr. Robert C. Binning serves as MRC Program Manager in this overall program entitled "Source Assessment," which includes the investigation of sources in each of four categories: combustion, organic materials, inorganic materials, and open sources. Dr. Dale A. Denny of the Industrial Processes Division at Research Triangle Park serves as EPA Project Officer. Reports prepared in this program are of three types: Source Assessment Documents, State-of-the-Art Reports, and Special Project Reports.

Source Assessment Documents contain data on emissions from specific industries. Such data are gathered from the literature, government agencies, and cooperating companies. Sampling and analysis are also performed by the contractor when the available information does not adequately characterize the source emissions. These documents contain all of the information necessary for IERL to decide whether emissions reduction is required.

State-of-the-Art Reports include data on emissions from specific industries which are also gathered from the literature, government agencies, and cooperating companies. However, no extensive sampling is conduced by the contractor for such industries. Results from such studies are published as State-of-the-Art Reports for potential utility by the government, industry, and others having specific needs and interests.

Special projects provide specific information or services which are applicable to a number of source types or have special utility to EPA but are not part of a particular source assessment study. This special project report, "Source Assessment: Textile Plant Wastewater Toxics Study, Phase II," was prepared to examine the level of toxicity (as measured by results of bioassay tests) and specific toxic pollutant removal attained by selected tertiary treatment systems treating secondary effluents from textile plants. Dr. Max Samfield of the Chemical Processes Branch at IERL-RTP served as EPA Task Officer.

The initial report in the project, "Source Assessment: Textile Plant Wastewater Toxics Study, Phase I" (EPA 600/2-78-004h), was prepared to provide chemical and toxicological data on secondary influent and effluent at 23 textile plants, and it served as a screening study for Phase II.

ABSTRACT

This study, sponsored by the EPA, was concerned with BATEA for the textile manufacturing industry. The purpose was to examine the level of removal of specific toxic pollutants and toxicity (as measured by results of bioassay tests) attained by selected tertiary systems treating secondary effluents from textile plants. Tertiary treatment systems consisting of unit processes arranged in various ways were ranked according to their apparent capabilities for removal of specific toxic pollutants and toxicity. The unit processes employed included flocculation/sedimentation, multimedia filtration with and without precoagulation, granular activated carbon adsorption, and ozonation. These unit operations were mobile and, thus, taken to numerous textile plants for evaluation.

The assessment of the treatment systems was based on both specific toxic pollutant analysis data and bioassay data gathered at eight textile plant locations where the treatment systems were tested. Samples collected from secondary and tertiary effluent streams were analyzed for specific toxic pollutants and submitted for the following bioassay analyses: freshwater ecology series (fathead minnow, bluegill, *Daphnia*, and algae), microbial mutagenicity, and cytotoxicity. For comparison, plant intake waters were also analyzed for specific toxic pollutants. Based on apparent specific toxic pollutant removal and toxicity removal, multimedia filtration-activated carbon was the best system. Tertiary treatment systems whose effluents contained a high level of residual coagulant appeared to be more detrimental to water quality, based on bioassay tests, than those systems which did not use coagulants. In addition, ozonation appeared to add toxic metals to the wastewater being treated.

This report was submitted in partial fulfillment of Contract 68-02-1874 by Monsanto Research Corporation under the sponsorship of the U.S. Environmental Protection Agency. This report covers a period from August 1977 to May 1979.

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ABBREVIATIONS

AA -- atomic absorption
ATMI -- American Textile Manufacturers Institute
BATEA -- best available technology economically achievable
BOD$_5$ -- 5-day biochemical oxygen demand, mg/ℓ
CHO-K1 -- Chinese Hampster Ovary - designation K-1
COD -- chemical oxygen demand, mg/ℓ
DNA -- deoxyribonucleic acid
EC -- electron capture detector on a gas chromatograph
EC$_{50}$ -- effective concentration at which 50% of the test species reach the desired effect, % effluent
EPA -- Environmental Protection Agency
GC -- gas chromatograph
GC/MS -- gas chromatography/mass spectroscopy
HLI -- Howard Laboratories, Incorporated
ICAP -- inductively coupled argon plasma
K-D -- Kuderna-Danish
LC$_{50}$ -- lethal concentration which causes 50% mortality in the test species, % effluent
MRC -- Monsanto Research Corporation
MS -- mass spectrometer
PCB -- polychlorinated biphenyls
PCS -- Pollution Control Science
SIM -- selected ion mode
TDS -- total dissolved solids, mg/ℓ
TSS -- total suspended solids, mg/ℓ

SECTION 1

INTRODUCTION

The Industrial Environmental Research Laboratory - RTP (IERL/RTP) of the U.S. Environmental Protection Agency (EPA) is currently engaged in a joint study with the American Textile Manufacturers Institute (ATMI; EPA Grant No. 804329) to determine the best available technology economically achievable (BATEA) for textile plant wastewaters. A total of 23 textile mills representing 8 textile processing categories and having well-operated secondary wastewater treatment facilities were selected by EPA and ATMI for the BATEA study. For that study, 2 mobile wastewater treatment pilot plants were constructed to gather technical data to identify the best available technology applicable to the 23 plants. The grant study focused on only a limited number of so-called criteria pollutants; i.e., 5-day biochemical oxygen demand, chemical oxygen demand, color, sulfides, total suspended solids, phenol, and pH.

On 7 June 1976 the U.S. District Court of Washington, D.C., issued a consent decree (resulting from Natural Resources Defense Council, et al. vs. Train) requiring EPA to enhance development of effluent standards. The court mandate focused federal water pollution control efforts on potentially toxic and hazardous pollutants. In response to the consent decree EPA developed a list of 129 specific compounds (known either as toxic or priority pollutants) that the agency agreed to consider during the standards setting process. Based on the consent decree, EPA-IERL/RTP decided to conduct a study parallel to the ATMI/EPA Grant Study of the textile industry. The objective of the IERL/RTP study was to determine both the removal efficiencies for the 129 consent decree specific toxic pollutants and the reduction in toxicity, as measured by bioassay tests, by the tertiary treatment technologies being investigated under the original grant study. Monsanto Research Corporation (MRC) conducted this toxics study under contract 68-02-1874.

The overall wastewater toxicity study, was divided into two phases. The first, covered by an initial MRC report, "Source Assessment: Textile Plant Wastewater Toxics Study, Phase I" (EPA 600/2-78-004h), established a baseline data base concerning toxicity and level of toxic pollutants present in raw wastewater and secondary effluents at 23 textile plants. The data were used to screen the 23 plants and to select those plants with secondary effluents of highest toxicity for further study under Phase II. Based on recommendations by the EPA IERL/HERL - RTP Bioassay Subcommittee, the following ten plants were recommended for Phase II study (listed in order of decreasing toxicity): N, A, W, C, T, V, L, S, P, and R. As the ATMI/EPA Grant Study proceded, two of the ten plants (Plants L and R) were dropped from the program due to scheduling difficulties, and thus were eliminated from the MRC Phase II scope of work.

The second phase of the effort, covered in this report, determined the reduction in specific toxic pollutant concentrations and in toxicity by the mobile pilot plant tertiary treatment systems. Only those plants selected in the first phase of the study were investigated.

Covering the second phase of the toxics study, this report describes sampling, chemical analysis, and bioassay procedures used. Chemical analyses of intake water, secondary effluents, and tertiary effluents are presented for the eight locations visited by the mobile pilot plants. Bioassay data are presented only for secondary and tertiary effluents.

The tertiary treatment systems are ranked according to their apparent specific toxic pollutant and toxicity removal capabilities. Other trends apparent in the data are also discussed.

Another activity associated with Phase II involved the Phase I organic data. Analysis of the organic compounds in the secondary effluent of the Phase I samples indicated that the organic priority pollutant species only accounted for 1% to 3% by weight of the total mass of organics. Therefore, EPA decided to reexamine the original instrumental data, which were stored on magnetic tape, and to identify as many of the other organic compounds as possible. This report also discusses the results of that study.

SECTION 2

SUMMARY

The purpose of this Phase II study was to determine reduction in toxicity (as measured by results of bioassay tests) and specific toxic pollutant concentrations achieved by the tertiary treatment systems under investigation in the ATMI/EPA Grant Study (Grant No. 804329). In this study, tertiary treatments of secondary effluents from eight textile plants in the United States were evaluated. In all, eight tertiary treatment systems as defined in Figure 1 were investigated. In the text of this report, treatment system types are identified by the type number defined in Figure 1. Samples of secondary effluent and effluents from each unit operation of the various tertiary treatment systems were analyzed for the 129 consent decree specific toxic pollutants and were also submitted for the following bioassay toxicity tests: freshwater ecology series (algae, *Daphnia*, fathead minnow and/or bluegill), microbial mutagenicity, and cytotoxicity. In addition, the samples were analyzed for various metals and nutrients not

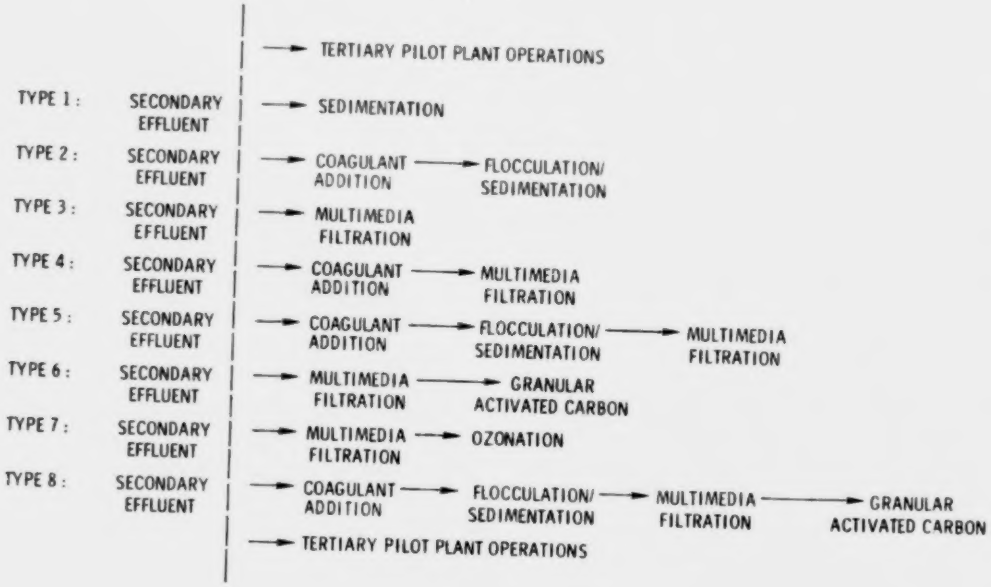
TERTIARY PILOT PLANT OPERATIONS
TYPE 1: SECONDARY EFFLUENT → SEDIMENTATION
TYPE 2: SECONDARY EFFLUENT → COAGULANT ADDITION → FLOCCULATION/ SEDIMENTATION
TYPE 3: SECONDARY EFFLUENT → MULTIMEDIA FILTRATION
TYPE 4: SECONDARY EFFLUENT → COAGULANT ADDITION → MULTIMEDIA FILTRATION
TYPE 5: SECONDARY EFFLUENT → COAGULANT ADDITION → FLOCCULATION/ SEDIMENTATION → MULTIMEDIA FILTRATION
TYPE 6: SECONDARY EFFLUENT → MULTIMEDIA FILTRATION → GRANULAR ACTIVATED CARBON
TYPE 7: SECONDARY EFFLUENT → MULTIMEDIA FILTRATION → OZONATION
TYPE 8: SECONDARY EFFLUENT → COAGULANT ADDITION → FLOCCULATION/ SEDIMENTATION → MULTIMEDIA FILTRATION → GRANULAR ACTIVATED CARBON
TERTIARY PILOT PLANT OPERATIONS

Figure 1. Tertiary treatment systems.

appearing on the consent decree toxic pollutant list. Samples of the intake water were collected at each textile plant and analyzed for the 129 consent decree specific toxic pollutants and various other metals, but they were not submitted for bioassay tests.

Separate composited samples of textile plant intake water, secondary effluent, and tertiary effluents were collected manually over a 24-hr period. Sampling began when the tertiary treatment system under investigation at each plant were operated under steady-state conditions. Samples were chemically preserved, stored in ice at 4°C, and shipped by air freight or delivered by the sampling crew to the various laboratories for appropriate chemical analyses and bioassay studies.

Most of the 129 specific toxic pollutants in textile plant intake water, secondary effluent, and tertiary effluent samples (totaling 44 samples) were analyzed by Monsanto Research Corporation (MRC). The EPA analytical protocol divides the 129 specific toxic pollutants into 5 fractions for analysis: volatile compounds, base/neutral compounds, acid compounds, pesticides and polychlorinated biphenyls (PCB's), and metals. EPA recommended that laboratories not acquire analytical standards for 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) because of its extreme toxicity. Asbestos was not analyzed due to the presence of interfering fibrous materials in textile wastewaters. Analytical procedures followed those recommended by EPA.

Seven organic toxic pollutants were found in the secondary effluent from at least one textile plant in concentrations greater than 10 μg/ℓ. The 10 μg/ℓ was selected as a concentration which is well above analytical detection limits and above probable levels of contamination. These include bis(2-ethylhexyl) phthalate, 1,2-dichlorobenzene, 1,2,4-trichlorobenzene, toluene, methylene chloride, di-n-butyl phthalate, and total phenol. However, the presence of bis(2-ethylhexyl) phthalate, methylene chloride, and di-n-butyl phthalate in the samples could have originated from contamination by the materials of which the pilot plant was constructed. Except for mercury, selenium, and thallium, all inorganic toxic pollutants were found in at least one textile plant secondary effluent in concentrations greater than 10 μg/ℓ (or the detection limit).

Secondary and tertiary effluent samples were also submitted for static bioassay studies. Table 1 briefly defines the purpose of each bioassay test used in the Phase II program. Procedures used in conducting the algal, *Daphnia*, and fish assays were based on protocols developed by the U.S. Environmental Protection Agency.

TABLE 1. BIOASSAY STUDIES CONDUCTED IN PHASE II

Bioassay test system	Indicator organisms	Purpose of test
Freshwater algal assay	*Selenastrum capricornutum*	To detect potential toxicity to aquatic plants.
Freshwater static bioassay	*Pimephales promelas* (fathead minnow) *Daphnia magna* (daphnid) *Lepomis macrochirus* (bluegill)	To detect potential toxicity to organisms in aquatic environments.
Microbial mutagenicity	*Salmonella typhimurium* (Ames test) (strains TA1535, TA1538, TA1538, TA98, TA100) *Escherichia coli* (pol A test) (strains W3110, p3478)	To determine if a chemical mutagen (possibly a carcinogen) is present. These microbial strains were selected because of their sensitivity to various classes of chemical compounds.
Cytotoxicity	Chinese hamster ovary cells	To measure metabolic impairment and death in mammalian cells. These primary cell cultures have some degree of metabolic repair capability.

Of the eight secondary effluents submitted for algal bioassay, six were toxic when compared to a control while five of the eight secondary effluents produced acute *Daphnia* toxicity in bioassay studies. Acute toxicity to bluegill fish was detected only twice although bluegill bioassays were conducted on only two of the eight secondary effluents, and acute toxicity to fathead minnows was detected with only three secondary effluents although only seven were tested.

Microbial mutagenicity was determined with both the Ames test and the pol A test. The Ames test uses mutant strains of *Salmonella* while the pol A test used in the Phase II study employs *Escherichia coli*. Cytotoxicity was determined using Chinese hamster ovary cells (CHO-K1). No mutagenicity or cytotoxicity was found in any of the secondary effluent or tertiary effluent streams tested.

The analytical and bioassay data were then used to identify positive and negative results which might be expected in applying various technologies to further treat secondary effluents from textile mills. In most cases, additional research would be required to adequately confirm the preliminary results identified in this study. Toxic organic analyses yielded little information regarding relative effectiveness of the various tertiary treatment systems in reducing toxic organic pollutant concentrations since the secondary effluent from only two plants contained toxic organic pollutants other than those which may have resulted from contamination, in concentrations greater than 10 $\mu g/\ell$.

Toxic metals were found to some extent in virtually all wastewater streams analyzed. Multimedia filtration, multimedia filtration followed by activated carbon adsorption, and flocculation/sedimentation followed by activated carbon adsorption were most effective in reducing levels of toxic metals. Multimedia filtration with precoagulation and ozonation proceeded by multimedia filtration were least effective in removing toxic metals. Toxic metals appeared to be added by ozonation.

Cyanide was effectively removed by multimedia filtration followed by both activated carbon adsorption and ozonation. Multimedia filtration alone was ineffective in removing cyanide.

Multimedia filtration and multimedia filtration followed by activated carbon adsorption appeared to be best at removing acute toxicity (as measured by bioassay tests). Results of the Phase II study also indicated that tertiary treatment systems that left high levels of residual aluminum or iron from coagulation in their effluents generally increased the toxicity of the wastewater, as compared with treatment systems whose effluents contained lower levels. In addition, systems employing cationic polymer coagulation were generally detrimental to water quality in terms of acute toxicity to algae.

The demonstrated capabilities of eight treatment systems to remove toxic pollutants and toxicity, as measured by bioassay tests, are listed as follows:

- Removal of Toxic Organic Compounds

  Insufficient data on which to base conclusions.

- Removal of Toxic Metals

  Best Removal Ability

  Multimedia filtration; flocculation/sedimentation followed by multimedia filtration; multimedia filtration followed by activated carbon

  Intermediate Removal Ability

  Sedimentation; flocculation/sedimentation; flocculation/sedimentation followed by multimedia filtration and activated carbon

  Least Removal Ability

  Multimedia filtration with precoagulation; multimedia filtration followed by ozonation

- Removal of Cyanide

  Best Removal Ability

  Multimedia filtration followed by activated carbon; multimedia filtration followed by ozonation

  Intermediate Removal Ability

  Sedimentation; flocculation/sedimentation; multimedia filtration with precoagulation; flocculation/sedimentation followed by multimedia filtration; flocculation/sedimentation followed by multimedia filtration and activated carbon

  Least Removal Ability

  Multimedia filtration

- Removal of Acute Toxicity

  Best Removal Ability

  Multimedia filtration; multimedia filtration followed by activated carbon

  Intermediate Removal Ability

  Sedimentation; flocculation/sedimentation followed by multimedia filtration; multimedia filtration followed by ozonation; flocculation/sedimentation followed by multimedia filtration and activated carbon

  Least Removal Ability

  Flocculation/sedimentation; multimedia filtration with precoagulation

- Composite

  Best Removal Ability

  Multimedia filtration followed by activated carbon

  Intermediate Removal Ability

  Sedimentation; flocculation/sedimentation; multimedia filtration; flocculation/sedimentation followed by multimedia filtration; multimedia filtration followed by ozonation; flocculation/sedimentation followed by multimedia filtration and activated carbon

  Least Removal Ability

  Multimedia filtration with precoagulation

The final activity of the Phase II study was to re-examine the gas chromatograph/mass spectrometer (GC/MS) spectra from the organic priority pollutant analysis of the Phase I secondary effluent samples, stored on magnetic tape, and identify as many organic compounds as possible.

The magnetic tapes were collected and individual sample spectra examined for major ion fragments. Using the elution time and three principal ions, specific organic compounds were identified. Their estimated percent abundance was calculated based on peak areas as compared to compounds of known concentration. It was not possible to quantify the concentrations because the GC/MS could not be set up to duplicate the instrumental conditions used 1 year earlier when the data were originally collected.

Analysis of the spectra found organic compounds common to the textile industry such as chlorobenzenes, C_9 to C_{20} aliphatic (paraffinic and olefinic) hydrocarbons, benzoic acid, soaps, and chlorophenols. Methylene chloride, found in moderate concentrations, was present principally due to laboratory contamination. Triphenyl phosphine, triphenyl phosphine oxide, and triphenyl phosphine sulfide, which are detergent derivatives, were detected in varying amounts and may be present due to residues on the sampling bottles or laboratory glassware. Further analysis of the data indicated some extraction carryover effects; i.e., the same organic species were found in both the base/neutral fraction and the acid fraction.

SECTION 3

RESULTS AND RECOMMENDATIONS

The data presented in this report can be used to identify preliminary positive and negative results which might be expected in applying various technologies to further treat secondary effluents from textile mills. In most cases, additional research would be required to adequately confirm these preliminary results obtained in this study. The preliminary results are summarized below:

1. Only seven organic toxic pollutants in excess of 10 μg/ℓ were seen in any of the secondary effluents of the eight textile plants. However, three of the compounds may have resulted from contamination.

2. Since few organic pollutants were presented in the wastewater streams, this data provided minimal unit operations performance information.

3. Eleven inorganic toxic pollutants were seen in at least one of the eight secondary effluents in levels greater than 10 μg/ℓ (or the detection limit).

4. Of the eight tertiary treatment systems tested, multi media filtration followed by granular activated carbon adsorption demonstrated the best overall toxic pollutant and toxicity removal capability, as determined by inorganic toxic pollutant analyses and bioassays.

5. Of the eight treatment systems tested, multimedia filtration with precoagulation demonstrated the worst over all toxic pollutant and toxicity removal capability, as determined by inorganic toxic pollutant analyses and bioassays.

6. Tertiary treatment systems that left high levels of residual inorganic coagulant in their effluents generally increased the toxicity of the wastewater, as compared with treatment systems whose effluents contained lower levels.

7. Systems employing cationic polymer coagulation increased the toxicity of the wastewater being treated, as measured by acute algal bioassays.

8. None of the secondary effluents or tertiary effluents gave a positive response in the mutagenicity or cytotoxicity tests.

9. The freshwater algal assay was the most sensitive bioassay test used in the Phase II program. The *Daphnia* assay was second most sensitive.

Based on the preliminary results listed above, the following recommendations can be made:

1. Coagulation as a tertiary tratement technology should be further investigated in terms of its effect upon water quality, as measured by bioassays. It appears that when the coagulant is not adequately removed from the wastewater stream following its use, water quality, as measued by bioassays, is adversely affected.

2. Since freshwater algal and *Daphnia* bioassays were the more sensitive bioassays, these tests should be considered first as a means to characterize the toxicity of textile mill wastewaters.

Based on the results of re-examining the Phase I priority pollutant data base, it is obvious that many organic compounds other than specific priority pollutants are present. Therefore, in a total environmental assessment study, the analytical scheme must have provisions for a more comprehensive analysis scheme than just looking for 129 specific compounds.

SECTION 4

SCOPE OF WORK

BACKGROUND

To understand the nature and purpose of the textile wastewater toxics program, it is first necessary to briefly review the events which led up to this study.

In June 1974 the U.S. Environmental Protection Agency's Effluent Guidelines Division set forth guidelines for the degree of effluent reduction attainable through the application of the "Best Practicable Control Technology Currently Available" and the "Best Available Technology Economically Achievable" which must be achieved by existing textile manufacturing (SIC 22) point sources by 1 July 1977 and 1 July 1983, respectively (1). However, on 1 October 1974 the textile manufacturing industry represented by the American Textile Manufacturers Institute, Northern Textiles Association, and Carpet and Rug Institute filed a petition with the U.S. Fourth Circuit Court of Appeals asking for a review of the proposed 1983 effluent guidelines. Grounds for the suit were that the BATEA had not been demonstrated for the textile manufacturing industry. As a result, ATMI and EPA filed a joint motion for delay of the petition, stating that additional information would be developed through a cooperative grant study by ATMI and EPA's Industrial Environmental Research Laboratory.

The objective of the ATMI/EPA Grant Study was to gather enough technical and economic data to determine the BATEA for reducing criteria pollutants from textile wastewaters. Criteria pollutants for the textile industry include 5-day biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), color, sulfide, pH, chromium, phenol, and total suspended solids (TSS).

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(1) Gallup, J. D. Development Document for Effluent Limitations Guidelines and New Source Performance Standard for the Textile Mills Point Source Category. EPA-440/1-74-022a (PB 238 832), U.S. Environmental Protection Agency, Washington, D.C., June 1974. 246 pp.

To evaluate the best available technology, two mobile pilot plants were constructed and operated by ATMI's contractor, Engineering Science, Inc. This stategy allowed for real-time, flowthrough treatability studies. Each pilot plant contained 5 tertiary wastewater treatment unit operations; 1 pilot plant was scheduled to visit 12 textile plants and the other to visit 11. An additional tertiary treatment technology was laboratory tested.

Treatment operations in each mobile unit include a reactor/clarifier (using combinations of alum, lime, ferric chloride, and anionic and cationic polyelectrolytes), two multimedia filters, three granular activated carbon columns, ozonation, and dissolved air flotation. Using these six operations, ATMI and EPA initially selected seven treatment modes for evaluation (Figure 2).

MODE A: REACTOR / CLARIFIER → MULTIMEDIA FILTER
MODE B: MULTIMEDIA FILTER → GRANULAR ACTIVATED CARBON COLUMNS
MODE C: MULTIMEDIA FILTER → OZONATOR
MODE D: OZONATOR
MODE E: (OPTIONAL) REACTOR / CLARIFIER → MULTIMEDIA FILTER → GRANULAR ACTIVATED CARBON → OZONATOR
MODE F: COAGULATION → MULTIMEDIA FILTER
MODE G: DISSOLVED AIR FLOTATION

Figure 2. Seven tertiary treatment modes initially selected for "best available technology" evaluation.

Each of the seven treatment modes was to be individually set up, and operational and pollutant data collected over a 2-day to 3-day period. Based on those data, the "best" system, referred to as the "candidate mode," was to be set up for 2 weeks of operational evaluation. These data were then to be forwarded for economic evaluation.

Prior to pilot plant field testing, a second EPA regulatory event occurred and formed the basis for the toxicity study. On 7 June 1976, the U.S. District Court of Washington, D.C., issued a consent decree (resulting from Natural Resources Defense Council et al. vs. Train) requiring EPA to accelerate development of effluent standards for 21 industrial point sources, including textile manufacturing. Among other requirements, the Court's mandate focused federal water pollution control on potentially toxic and hazardous chemical compounds. The original consent decree required that "65 classes" of chemical compounds be analyzed in wastewater samples. Recognizing the difficulty of analyzing for all chemical species present in each category of compounds, EPA developed a surrogate list of 129 specific compounds representative of the classes of compounds listed in the consent decree. These compounds are referred to as "toxic pollutants" or "priority pollutants" and are divided into several fractions for sampling and analytical purposes as shown in the appendix. EPA also developed a sampling and analytical procedures manual to be used as a laboratory guide for toxic pollutant assessment (2).

The consent decree obligates EPA to identify which toxic pollutants are present in industrial wastewaters and to determine the ability of various wastewater treatment technologies to remove toxic pollutants. Therefore, EPA with ATMI's cooperation decided to conduct a separate, but parallel, study with the EPA/ATMI Grant Study designed to measure toxic pollutants. Also, since the consent decree focused on the issue of wastewater toxicity, ATMI agreed to have samples collected for bioassay testing in order to have a complete and comprehensive wastewater characterization data base. Therefore, the bioassay testing program established by EPA for evaluating the reduction in toxicity of water samples by control technologies was integrated into the program (3). Thus, the overall EPA-IERL/RTP textile program consists of two separate projects, each with different activities, running parallel in time, but converging towards the same goal: determination of the best available technology economically achievable for textile wastewaters (Figure 3).

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(2) Draft Final Report: Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants. U.S. Environmental Protection Agency, Cincinnati, Ohio, April 1977. 145 pp.

(3) Duke, K. M., M. E. Davis, and A. J. Dennis. IERL-RTP Procedures Manual: Level 1 Environmental Assessment Biological Tests for Pilot Studies. EPA-600/7-77-043 (PB 268 484), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, April 1977. 114 pp.

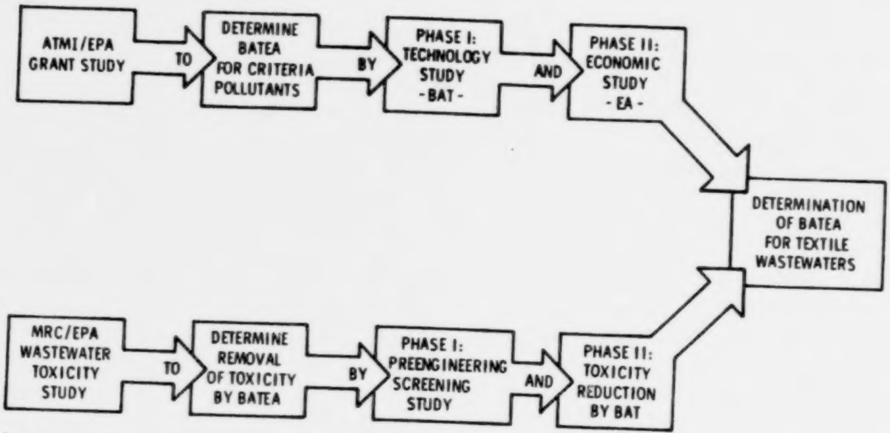
ATMI/EPA GRANT STUDY
TO
DETERMINE BATEA FOR CRITERIA POLLUTANTS
BY
PHASE I: TECHNOLOGY STUDY -BAT-
AND
PHASE II: ECONOMIC STUDY - EA -
DETERMINATION OF BATEA FOR TEXTILE WASTEWATERS
MRC/EPA WASTEWATER TOXICITY STUDY
TO
DETERMINE REMOVAL OF TOXICITY BY BATEA
BY
PHASE I: PREENGINEERING SCREENING STUDY
AND
PHASE II: TOXICITY REDUCTION BY BAT

Figure 3. Overall program approach to determine BATEA.

PROGRAM OBJECTIVE

The fundamental objective of the textile wastewaters program conducted by MRC in conjunction with the EPA is to determine the reduction in toxicity and toxic pollutant concentrations achieved by the tertiary treatment systems under investigation in the ATMI/EPA Grant Study. To evaluate the reduction in toxicity in a cost-effective manner for the MRC/EPA project, a two-phase approach was developed. Phase I was designed to collect baseline toxicity and toxic pollutant data on secondary effluents from 23 selected textile plants and to rank the plants in descending order of toxicity. Data obtained in the Phase I study are reported in "Source Assessment: Textile Plant Wastewater Toxics Study, Phase I" (4). Phase II was designed to determine the level of toxicity and toxic pollutant removal attained by the tertiary treatment systems in the ATMI/EPA Grant Study at only those plants with relatively high secondary effluent toxicity, as determined in Phase I.

PROJECT ORGANIZATION - PHASE II

The major effort of the Phase II MRC/EPA tertiary treatment assessment study was devoted to the collection, chemical analysis, and biological toxicity testing of single, 24-hr composited wastewater samples from mobile tertiary treatment pilot plants located at eight[a] textile mills. In addition, samples of intake water were collected for chemical analysis.

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(4) Rawlings, G. D. Source Assessment: Textile Plant Wastewater Toxics Study, Phase I. EPA-600/2-78-004h, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, March 1978. 153 pp.

Under the Phase II program, wastewater samples were collected by MRC (for subsequent toxic pollutant analyses and bioassay) during the last 2 wk of pilot plant operation at each site. The pilot plants were operated by Engineering Sciences, Inc., who monitored certain criteria pollutants for BATEA evaluation of the tertiary treatment systems. During the MRC sampling period, the pilot plants were operated under steady-state conditions when "candidate" modes of operation were studied. "Candidate" modes of operation were defined as tertiary treatment systems, each consisting of various treatment technologies, which appeared to be most promising in improving secondary effluent quality to meet suggested BATEA guideines, as determined by Engineering Science, Inc., based on initial screening studies conducted prior to the last two weeks of operation.

The MRC wastewater samples were shipped immediately after collection to various analytical and bioassay laboratories, including MRC's Dayton Laboratory. Laboratories involved in the analysis and bioassay work are discussed in Section 6 and 7. Data from chemical analyses and bioassays were then assembled and evaluated by MRC. Both phases of the textile wastewater toxics study were directed by Dr. Max Samfield of the Chemical Processes Branch, IERL/RTP, EPA. For the Phase II program, Dr. Gary D. Rawlings of MRC served as contract manager and Mr. Jeffrey R. Klieve of MRC served as principal investigator.

SPECTRA ANALYSIS

For this portion of the project, magnetic tapes storing all the chromatograms of the Phase I secondary effluent samples were collected and loaded on the HP 5934 Data System. Individual samples were then accessed and the total ion chromatograph for each was displayed on the data system screen.

The major compound identification search strategy involved accessing major ions and determining when (scan number) they eluted from the GC during the 40-min run for each compound. When the elution time was identified, the computer would display all ions produced at that time. Using the three major ions and their relative intensities, the compound was identified by data listed in Reference 5.

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[a] After completion of Phase I, 10 textile mill sites were recommended for Phase II studies. Two of the recommended sites were eliminated from the ATMI/EPA Grant Study, and thus from Phase II of the MRC/EPA wastewater toxics study. Therefore, the Phase II toxics study was conducted at only eight sites.

The estimated percent abundance of the compound was determined based on the peak areas of the dominant ion fragments and does not take into account different response ratios for different organic compounds. The data serve as an indication of bulk composition.

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(5) Eight Peak Index of Mass Spectra, Vol. III, Second Edition, Table 3 (Part 1). Mass Spectrometry Data Centre, AWRE, Aldermaston, Reading, United Kingdon, 1974. 1933 pp.

SECTION 5

SAMPLING PROCEDURES

Samples of intake water, secondary effluent, and various tertiary effluents were collected for subsequent toxic pollutant analyses and bioassay at each of the eight sites of the Phase II toxics study. The sampling was conducted by MRC personnel during one 24-hr period occurring during the last 2 wk of pilot plant operation. During these last 2 wk, the pilot plant was operated under steady-state conditions when pilot plant studies were conducted to assess "candidate" modes of operation. Selected unit operations were generally run in series during this period. Prior to the last 2 wk, the unit operations contained in the pilot plant trailer had been operated individually for short periods of time in order to screen the unit processes for further study during the candidate phase.

For analyses other than volatile toxic pollutants, sets of time-proportional, manually composited samples were taken over a 24-hr period from each water stream subjected to the MRC sampling/analysis program. Aliquots making up composite samples of secondary and tertiary effluents were taken once per hour, while aliquots making up composite samples of intake water were generally taken every 3 hr. Therefore, each composite sample of secondary or tertiary effluent was made of 24 aliquots of equal size, and each composite sample of intake water was generally made of 8 aliquots of equal size.

For volatile toxic pollutant analyses, three grab samples were taken from each effluent stream. Each of the grab samples was hermetically sealed immediately after sample collection. Generally, one grab sample of each stream was taken during each of the three working shifts (8 a.m. to 4 p.m., 4 p.m. to 12 p.m., 12 p.m. to 8 a.m.). The three individual samples were then composited in the laboratory for a single analysis.

In collecting the samples extreme care was taken to assure the samples did not come into contact with materials which might induce contamination. At each point, a separate 3-gal Teflon®-lined stainless steel bucket or 1-ℓ glass beaker was used to collect the aliquot. The aliquot was then transferred to a 5-gal glass jug which was filled over a 3-hr period. Every 3 hr the sample collected in the jug was distributed to the various sample bottles listed in Table 2 (except samples for volatile

TABLE 2. REQUIRED SAMPLE CONTAINERS AND PRESERVATIVES

Analysis number	Analysis fraction	Container, per sampling point	Preservatives required
1	Volatile organics (including direct injectables)	Three 40-ml glass vials w/Teflon-lined septa	Keep at 4°C
2	Semivolatile organics (base/neutral, acid, pesticide, and PCB's), TSS, and color	One 4-ℓ amber glass pharmaceutical job w/Teflon-lined cap	Keep at 4°C
3	Metals	One 500-ml plastic bottle	In the lab, add 5 ml of redistilled nitric acid, keep at 4°C
4	Fish/*Daphnia* bioassay	Four to six 20-ℓ cubitainers	Keep at 4°C
5	Algae bioassay	One 20-ℓ cubitainer	Keep at 4°C
6	Microbiological bioassays	One 20-ℓ cubitainer of secondary effluent, One 1-ℓ amber glass bottle of other effluents	Keep at 4°C
7	Cyanide (total)	One 500-ml plastic bottle	Adjust pH ≥12 w/10N sodium hydroxide, keep at 4°C
8	Phenol (total) and COD	One 1-ℓ amber glass bottle	4 ml of conc sulfuric acid, keep at 4°C
9	Ammonia and nitrate	One 500-ml plastic bottle	2 ml of conc sulfuric acid, keep at 4°C
10	Total phosphate	One 500-ml plastic bottle	Keep at 4°C
11	Sulfide	One 500-ml plastic bottle	2 ml zinc acetate

organics analyses). Care was always taken to insure that the sample remained homogeneous while in the bucket, beaker, or jug. Proper preservatives (listed in Table 2) were added to the sample bottles immediately after the distribution from the 5-gal jug (i.e., 2 hr after the first aliquot was taken).

Prior to sampling, all glass sample bottles were cleaned thoroughly with strong acid (50% sulfuric acid + 50% nitric acid), rinsed with distilled water, and heated in a glass annealing oven at 400°C for at least 30 min. Once the glass bottles cooled to room temperature, Teflon-lined caps were applied. Plastic bottles were cleaned thoroughly by washing in nitric acid and rinsing several times with distilled water. Once in the field, all containers were rinsed with the appropriate sample water. Labels as shown in Figure 4 were filled out and affixed to the appropriate sample bottles prior to sampling. Once applied to the bottle, the label was covered with clear tape to prevent damage from water.

Job ______________________________

Sample or Run No. ______________________

Sample Location ________________________

Type of Sample _________________________

Analyze for ____________________________

Preservation ___________________________

Comments _______________________________

____

____

____

Log No. ________________ Date __________

Initials _______________________________

Figure 4. MRC bottle label used for sample identification.

At the completion of the 24-hr sampling effort, sample containers were checked to insure proper sample preservation, and bottle caps were sealed to the bottles to prevent sample leakage during shipment. All glass bottles were then wrapped with packing material. Sample containers were packed in ice chests filled with wet ice to maintain the sample temperature at 4°C. Sample chests were then transported to the appropriate laboratory for chemical analysis or bioassay either by commercial air freight or in the sampling van.

SECTION 6

CHEMICAL ANALYSIS PROCEDURES

INTRODUCTION

Chemical pollutants analyzed under the Phase II program included most of the pollutants on the toxic pollutant list (see the appendix) and other pollutants which were analyzed while analyzing for the toxic pollutants, analyzed to assess bioassay results, or analyzed as required by parallel studies. Various laboratories performed analyses during the program; they are shown in Figure 5. Analyses for criteria pollutants were performed for MRC by Pollution Control Science, Inc. (PCS) and Howard Laboratories, Inc. (HLI) in the Dayton, Ohio area, while most toxic pollutant analyses were conducted by MRC in-house.

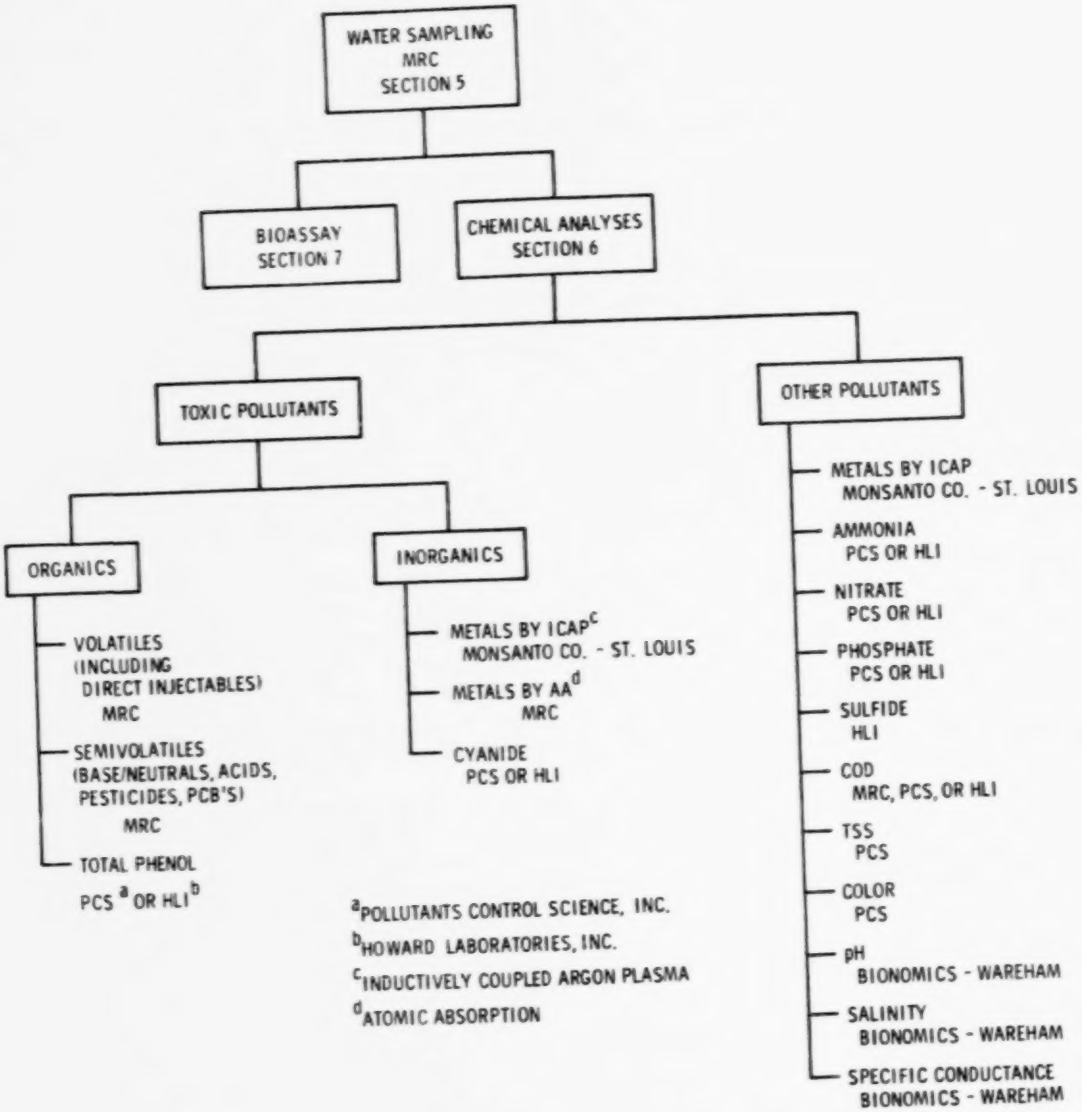
As shown in Figure 5, toxic pollutant compounds are categorized into a number of analysis fractions. The fractions include volatile compounds (including direct injectables), base/neutral extractable compounds, acid extractable compounds, pesticides, PCB's, total phenol, metals, and total cyanide. Base/neutral extractable compounds, acid extractable compounds, pesticides, and PCB's are grouped together as semivolatile compounds.

Still under development, procedures for the analysis of toxic pollutants require further verification and validation. Therefore, the data presented in Section 8, which were obtained by the procedures presented in this section, serve only to indicate toxic pollutant type and general concentrations.

ANALYTICAL PROCEDURES

Volatile Organic Toxic Pollutants

Toxic pollutants in this category were analyzed at the Dayton Laboratory of MRC by an analysis procedure designed by EPA to determine the concentration of those chemical species which are amenable to the Bellar purge and trap method (2). Sets of hermetically sealed 45-ml glass vials, which were collected from each of the Phase II sampling points, were composited in the laboratory and split into two fractions. One of the fractions was analyzed, and the other served as a backup sample. Figure 6 provides a simplified diagram of the analytical scheme used for volatile organics analysis in the Phase II program.


WATER SAMPLING
MRC
SECTION 5
BIOASSAY
SECTION 7
CHEMICAL ANALYSES
SECTION 6
TOXIC POLLUTANTS
OTHER POLLUTANTS
ORGANICS
INORGANICS
VOLATILES (INCLUDING DIRECT INJECTABLES)
MRC
SEMIVOLATILES (BASE/NEUTRALS, ACIDS, PESTICIDES, PCB'S)
MRC
TOTAL PHENOL
PCS[a] OR HLI[b]
METALS BY ICAP[c]
MONSANTO CO. - ST. LOUIS
METALS BY AA[d]
MRC
CYANIDE
PCS OR HLI
METALS BY ICAP
MONSANTO CO. - ST. LOUIS
AMMONIA
PCS OR HLI
NITRATE
PCS OR HLI
PHOSPHATE
PCS OR HLI
SULFIDE
HLI
COD
MRC, PCS, OR HLI
TSS
PCS
COLOR
PCS
pH
BIONOMICS - WAREHAM
SALINITY
BIONOMICS - WAREHAM
SPECIFIC CONDUCTANCE
BIONOMICS - WAREHAM

[a] POLLUTANTS CONTROL SCIENCE, INC.

[b] HOWARD LABORATORIES, INC.

[c] INDUCTIVELY COUPLED ARGON PLASMA

[d] ATOMIC ABSORPTION

Figure 5. Chemical analysis logistics - laboratories and analysis procedures index.

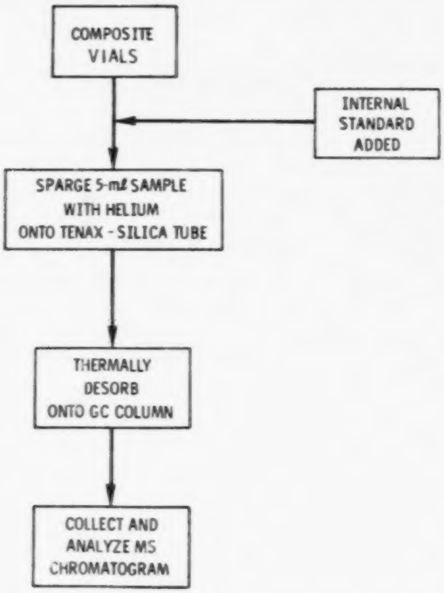

COMPOSITE VIALS
INTERNAL STANDARD ADDED
SPARGE 5-mℓ SAMPLE WITH HELIUM ONTO TENAX - SILICA TUBE
THERMALLY DESORB ONTO GC COLUMN
COLLECT AND ANALYZE MS CHROMATOGRAM

Figure 6. Analytical scheme for volatile organics analysis.

An internal standard, 1,4-dichlorobutane, was added to 5 ml of the composited wastewater sample, which was subsequently sparged with helium onto a Tenax-GC silica-packed sample tube.

Analyses were completed using a Hewlett-Packard 5981 gas chromatograph/mass spectrometer (GC/MS) with a 5934 data system. Sample tubes were heated to 180°C over a 1-min period. They were held at that temperature for 4 min to desorb the compounds onto a Carbowax 1500 column held at -40°C. For compounds with boiling points below room temperature, cryogenic trapping at -4°C (liquid nitrogen cooling) provided better reproducibility of retention time that did the suggested protocol temperature of 30°C. After desorption, the GC column temperature was raised at 8°C/min to 170°C.

Qualitative identification of a compound was made using the three criteria listed in the protocol (2): 1) retention time must coincide with known retention times, 2) three characteristic masses must elute simultaneously, and 3) intensities of the characteristic masses must stand in the known proper proportions. Quantitation of volatile organics was made using response ratios to the 1,4-dichlorobutane internal standard.

Direct injectables (acrolein and acrylonitrile) were analyzed by injecting a 5-μℓ aliquot of the volatile organics sample directly onto a Tenax-GC column of a gas chromatograph interfaced with a computer-controlled quadrupile mass spectrometer. The GC/MS were used in the selected-ion mode (SIM) to provide selectivity and enhanced sensitivity.

Semivolatile Organic Toxic Pollutants

Semivolatile organic toxic pollutants were also analyzed at MRC-Dayton Laboratory with procedures designed by EPA (2). Figure 7 depicts the sample processing scheme used for analyzing the base/neutral and acid fractions. Two liters of the wastewater sample were made alkaline (pH >11) using sodium hydroxide, then extracted three times using methylene chloride. The remaining aqueous phase was collected and acidified (pH <2) with hydrochloric acid, after which the organic acids were extracted with methylene chloride. Both methylene chloride extracts (one containing base/neutral organics, and the other containing organic acids) were dried on a column of anhydrous sodium sulfate. The dried extracts were then concentrated to 1.0 mℓ with a Kuderna-Danish (K-D) evaporator. Resultant solutions were spiked with deuterated anthracene, sealed in septum-capped vials, and stored at 4°C until analyzed. Gas chromatographic/mass spectrometric analyses were performed using SP 2250 and Tenax-GC columns for base/neutral and acid samples, respectively (2).

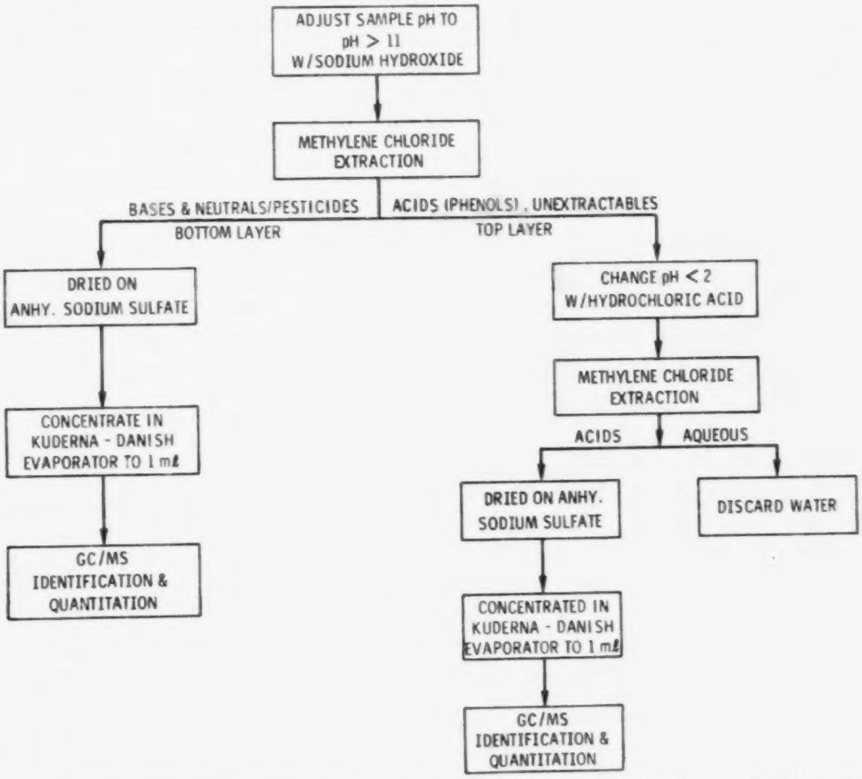

ADJUST SAMPLE pH TO pH > 11 W/SODIUM HYDROXIDE
METHYLENE CHLORIDE EXTRACTION
BASES & NEUTRALS/PESTICIDES
BOTTOM LAYER
ACIDS (PHENOLS), UNEXTRACTABLES
TOP LAYER
DRIED ON ANHY. SODIUM SULFATE
CONCENTRATE IN KUDERNA - DANISH EVAPORATOR TO 1 mℓ
GC/MS IDENTIFICATION & QUANTITATION
CHANGE pH < 2 W/HYDROCHLORIC ACID
METHYLENE CHLORIDE EXTRACTION
ACIDS
AQUEOUS
DRIED ON ANHY. SODIUM SULFATE
DISCARD WATER
CONCENTRATED IN KUDERNA - DANISH EVAPORATOR TO 1 mℓ
GC/MS IDENTIFICATION & QUANTITATION

Figure 7. Sample processing scheme for the analysis of the base/neutral and acid fractions of semivolatile organics.

The sample processing scheme used for pesticides and PCB's is shown in Figure 8. One liter of the wastewater sample was adjusted to a pH of 6.5 to 7.5 using 50% sodium hydroxide or concentrated hydrochloric acid. Pesticides and PCB's were extracted into a 15% methylene chloride/85% hexane solution which was separated and back extracted with acetonitrile to remove interfering compounds. Acetonitrile extract was chromatographed on two SP 2250 columns employing electron capture (EC) detection. Verification of pesticides and PCB's present was effected using an SP 2100 column.

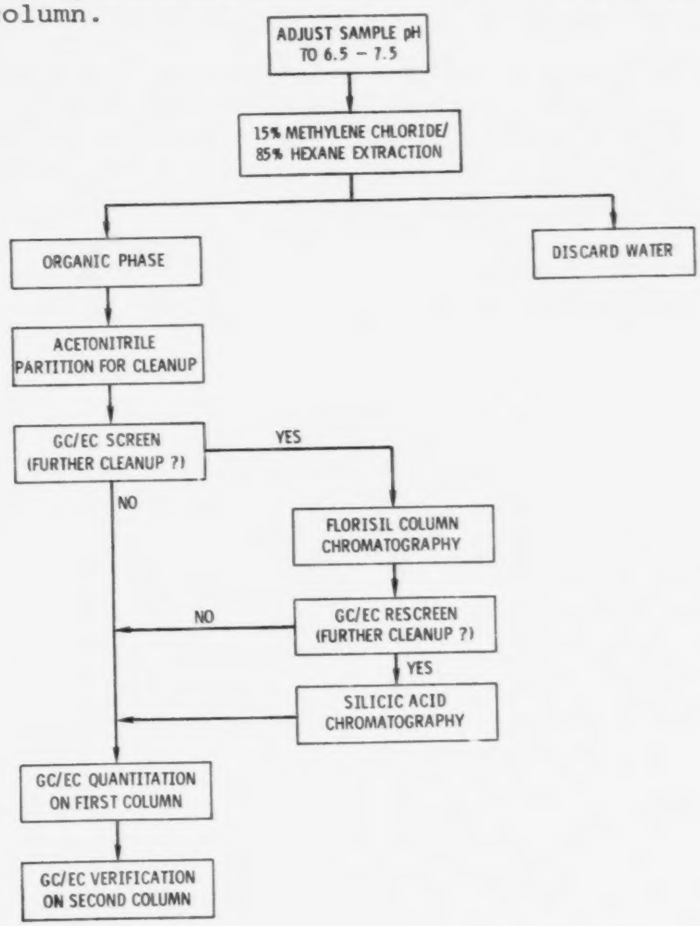

ADJUST SAMPLE pH TO 6.5 – 7.5
15% METHYLENE CHLORIDE/ 85% HEXANE EXTRACTION
ORGANIC PHASE
DISCARD WATER
ACETONITRILE PARTITION FOR CLEANUP
GC/EC SCREEN (FURTHER CLEANUP ?)
YES
NO
FLORISIL COLUMN CHROMATOGRAPHY
GC/EC RESCREEN (FURTHER CLEANUP ?)
NO
YES
SILICIC ACID CHROMATOGRAPHY
GC/EC QUANTITATION ON FIRST COLUMN
GC/EC VERIFICATION ON SECOND COLUMN

Figure 8. Sample processing scheme for pesticide and PCB analysis.

Total Phenol

In addition to specific phenolic compounds analyzed in the semivolatile toxic pollutant analysis scheme, total phenol was also measured. Determinations of total phenol concentrations were made by two laboratories during the Phase II program; Pollution Control Science, Inc. of Miamisburg, Ohio made the determinations in the early stages of the program, and Howard Laboratories, Inc. of Dayton, Ohio made them in the latter stages. Both laboratories used the chloroform extraction method following distillation [Standard Methods 510A and 510B (5)].

Inorganic Toxic Pollutants (Including Cyanide)

In addition to organic toxic pollutants, the toxic pollutant list given in the appendix includes 13 metals (measured as the total metal), total cyanide, and asbestos.

The following nine toxic pollutant metals were analyzed by Monsanto Company in St. Louis, Missouri by the ICAP excitation technique: antimony, beryllium, cadmium, chromium, copper, lead, nickel, silver, and zinc. Via the ICAP technique, simultaneous multielement determinations are made of trace metal concentrations at the sub-mg/ℓ level in solutions. The basis of this method is atomic emission. Excitation energy is supplied by coupling a nebulized sample with high temperature argon gas which has been passed through a powerful radio-frequency field. Emitted light is simultaneously monitored at 22 wavelengths corresponding to 22 different elements.

Since four of the toxic pollutant metals are not amenable to analysis by ICAP techniques, they were analyzed by standard atomic absorption (AA) techniques at MRC's Dayton Laboratory. The four metals are arsenic, mercury, selenium, and thallium. Procedures for AA analysis of these four metals are found in Standard Method 301A (6). Arsenic alone was analyzed during early stages of the Phase II program, since mercury, selenium, and thallium were not found in samples resulting from Phase I investigations (4). However, a parallel program to Phase II, undertaken part way through the Phase II study, required analysis of the other three AA metals, and thus the analytical results for mercury, selenium, and thallium are available for later pilot plant sites investigated in the Phase II study.

Total cyanide was analyzed using the colorimetric method following distillation [Standard Methods 413B and 413D (5)] by Pollution Control Science, Inc. of Miamisburg, Ohio during initial stages of the Phase II program, and by Howard Laboratories, Inc. of Dayton, Ohio during the later stages.

As in Phase I of the program asbestos was not analyzed in the Phase II program.

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(6) Standard Methods for the Examination of Water and Wastewater, Fourteenth Edition. American Public Health Association, Washington, D.C., 1976. 874 pp.

Other Pollutants

As mentioned earlier, pollutants other than toxic pollutants were analyzed in many cases during the Phase II program. Some analyses are available when they were cogenerated with some toxic pollutant analyses. Metals analyzed by ICAP which do not appear on the toxic pollutant list given in the appendix are examples. Some analyses (ammonia, nitrate, phosphate) were conducted to aid in interpreting bioassay results. Salinity, pH, and specific conductance were determined in conjunction with bioassay studies by EG&G International, Inc., while other analyses (COD, TSS, color, pH, sulfide) were required for studies parallel to the Phase II study.

Metals were determined via the ICAP technique by Monsanto Company in St. Louis, Missouri as discussed previously in this section. Table 3 (1, 6, 7) exhibits methodologies and laboratories used to conduct the other analyses.

---

(7) Manual of Methods for Chemical Analysis of Water and Wastes. EPA-625/6-76-003a (PB 259 973), U.S. Environmental Protection Agency, Cincinnati, Ohio, 1976. 317 pp.

TABLE 3. PROCEDURE USED IN ANALYZING OTHER POLLUTANTS

Analysis	Pollution Control Science, Inc.		Howard Laboratories, Inc.		EG&G, Bionomics Aquatic Toxicology Laboratory	
	Method	Reference	Method	Reference	Method	Reference
Ammonia	Nesslerization preceded by distillation	Standard Methods 418A and 418D (6)	Selective ion electrode method	Methods for Chemical Analysis of Water and Wastes, p. 165 (6)		
Nitrate	Brucine method	Standard Method 419D (6)	Nitrate electrode method	Standard Method 419B (6)		
Phosphate	Ascorbic acid method	Standard Method 425F (6)	Stannous chloride method preceded by persulfate digestion	Standard Methods 425CIII and 425E (6)		
Sulfide			Orion direct measurement method, model 94-16 electrode			
COD			Oxygen demand (chemical)	Standard Method 508 (6)		
TSS	Total nonfilterable residue dried at 103-105°C	Standard Method 208C (6)				
Color (ADMI)	ADMI color value	Development Document for Effluent Limitations Guidelines and New Source Performance Standards for Textile Mills-Appendix A (1)				
pH					Glass electrode technique	Standard Method 424 (6)
Salinity					Refractometry with American Optical refractometer	
Specific conductivity					Conductivity with Yellow Springs instrument model 33	Standard Method 205 (6)

SECTION 7

BIOASSAY PROCEDURES

INTRODUCTION

In addition to chemical analyses, a series of bioassay tests were conducted to assess toxicity removal capabilities of the tertiary treatment systems. In toxicity testing, organisms will integrate the synergistic and antagonistic effects of all the effluent components over the duration of exposure. Therefore, analyses of specific components cannot totally be related to toxicity, and toxicity testing must be performed. In Phase II, acute toxicity to various freshwater animal and plant species, microbiological mutagenicity, and cytotoxicity were determined. Table 1, repeated here for reader convenience, briefly defines the purpose of each of the bioassay tests. The laboratories performing various bioassay tests through the Phase II program are shown in Figure 9.

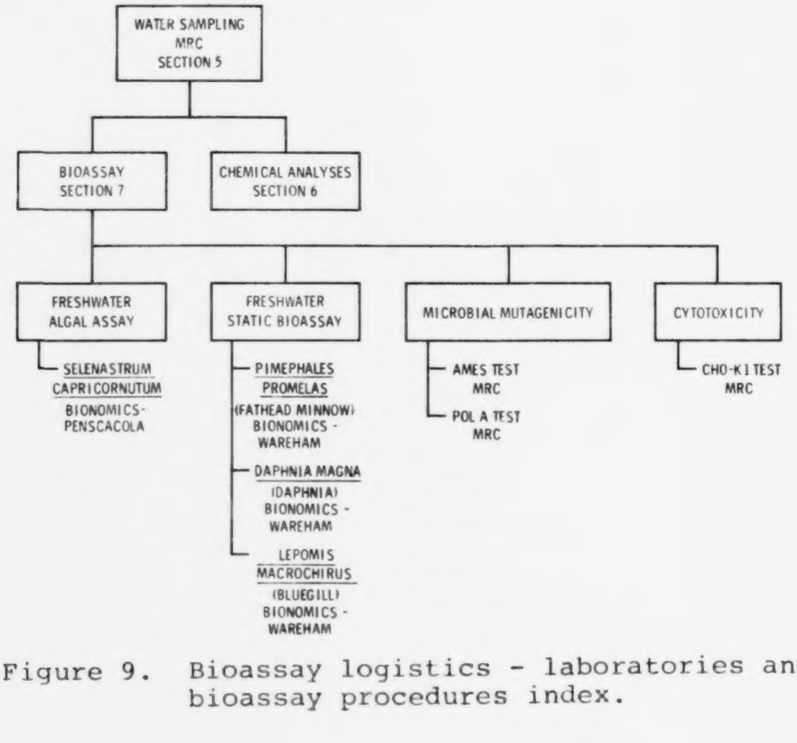
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Figure 9. Bioassay logistics - laboratories and bioassay procedures index.

TABLE 1. BIOASSAY STUDIES CONDUCTED IN PHASE II

Bioassay test system	Indicator organisms	Purpose of test
Freshwater algal assay	*Selenastrum capricornutum*	To detect potential toxicity to aquatic plants.
Freshwater static bioassay	*Pimephales promelas* (fathead minnow) *Daphnia magna* (daphnid) *Lepomis macrochirus* (bluegill)	To detect potential toxicity to organisms in aquatic environments.
Microbial mutagenicity	*Salmonella typhimurium* (Ames test) (strains TA1535, TA1538, TA1538, TA98, TA100) *Escherichia coli* (pol A test) (strains W3110, p3478)	To determine if a chemical mutagen (possibly a carcinogen) is present. These microbial strains were selected because of their sensitivity to various classes of chemical compounds.
Cytotoxicity	Chinese hamster ovary cells	To measure metabolic impairment and death in mammalian cells. These primary cell cultures have some degree of metabolic repair capability.

Acute toxicity tests were used in Phase II to determine the level of toxic agent in secondary or tertiary effluent that produced an adverse effect on a specified percentage of test organisms (algae, fathead minnow, bluegill, or *Daphnia*) in a short period of time. The most common acute toxicity test is the acute mortality test. Experimentally, 50% effect is the most reproducible measure of the toxicity of a toxic agent to a group of test organisms during a convenient, reasonably useful exposure time period. Thus, the acute mortality test is a statistical estimate of the LC_{50}, which is the median concentration of toxicant in dilution water, that is lethal to 50% of the test organisms during continuous exposure for a specified period of time. However, the effective concentration (EC_{50}), the concentration at which growth rate was 50% of a control, was determined when algae were used. The terms median lethal concentration (LC_{50}) and median effective concentration (EC_{50}) are consistent with the widely used terms median lethal dose (LD_{50}) and median effective dose (ED_{50}), respectively. "Concentration" refers to the amount of toxicant per unit volume of test solution; "dose" refers to the measured amount of toxicant given to the test organism.

Microbial mutagenicity bioassays were used in Phase II to determine if chemical mutagens were present in the secondary and tertiary effluents. Two bioassays were used to test these effluents. The *Salmonella typhimurium* assay (Ames test), using the five strains TA98, TA100, TA1535, TA1537, and TA1538, and the *Escherichia coli* pol $A^{+/-}$ assay using the p3478 and w3110 strains, were used by MRC to evaluate the effluents. Both assays included the Aroclor 1254-induced rat liver S-9 fraction for metabolic activation.

The *S. typhimurium* assay (Ames test) has proven to be 85% to 90% accurate in detecting mutagens (8). The assay as developed by Dr. Bruce N. Ames (9) uses the mutant strains of *Salmonella* which are histidine dependent. A minimal amount of histidine is added to the system to allow a background growth of the bacteria. If a mutation occurs, the bacteria revert back to the wild type, a histidine-independent strain, and these revertants grow into visible colonies during incubations. A test is then scored by counting the number of spontaneous revertant colonies on the control plates and the plate treated with the sample. A sample is considered to be mutagenic if the increase in the number of revertants on the plate with samples is at least two times that of the control and a dose response is exhibited.

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(8) McCann, J., E. Choi, E. Yamasaki, and B. N. Ames. Detection of Carcinogens as Mutagens in the *Salmonella*/Microsome Test: Assay of 300 Chemicals. Proceedings of the National Academy of Science, 72:5135-5139, 1975.

(9) Ames, B. N., J. McCann, and E. Yamasaki. Methods for Detecting Carcinogens and Mutagens with the *Salmonella*/Mammalian-Microsome Mutagenicity Test. Mutation Research, 31:347-364, 1975.

The *E. coli* pol $A^{+/-}$ assay as described by Slater, et al. (10) determines DNA damage by the use of the w3110 and p3478 strains, which are DNA repair-proficient and -deficient, respectively. Substances that affect the DNA of the *E. coli* inhibit the growth of the repair-deficient strain. This results in a zone of inhibition or no growth, whereas the proficient strain shows no inhibition. Some compounds may be toxic to both strains, resulting in zones of inhibition with both strains. The test is then scored by comparison of the inhibition of the two strains.

Cytotoxicity (cell toxicity) assays were performed to measure quantitatively any cellular metabolic impairment and death resulting from exposure *in vitro* to secondary and tertiary effluent samples. The clonal cytotoxicity assay for acute toxicity using Chinese hamster ovary cells (CHO-K1) was selected to test the secondary and tertiary effluents. MRC has developed this *in vitro* clonal assay using the CHO-K1 cells (11). This assay was used because of the sensitivity and reproducibility of the system.

The CHO cytotoxicity assay determines acute toxicity through the number of colonies formed after a 6-day incubation with the sample. A precise number of cells are plated, and only those cells that replicate into visible colonies are scored as survivors. The number of colonies of the control flask is compared to the number on the flask with sample, and the percent survival is determined.

PROCEDURES

Algal Assay

Immediately after collection, effluent samples were shipped to EG&G, Bionomics Marine Research Laboratory in Pensacola, Florida, where they were stored at 4°C until testing began. Culture and test procedures generally followed protocols developed by the the EPA (3).

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(10) Slater, E. E., M. D. Anderson, and H. S. Rosenkranz. Rapid Detection of Mutagens and Carcinogens. Cancer Research, 31:970-973, 1971.

(11) Wininger, M. T., F. A. Kulik, and W. D. Ross. *In Vitro* Clonal Cytotoxicity Assay Using Chinese Hamster Ovary Cells (CHO-K1) for Testing Environmental Chemicals. *In Vitro*, 14(4):381, 1978.

An inoculum of *Selenastrum capricornutum* was added to each of the test flasks, which contained varying concentrations of the secondary or tertiary effluents in dilution water. The test flasks were then incubated at 24 ± 1°C under 4,300 lux illumination. After 7, 12, and 14 days of exposure, a portion of the algal suspension was removed from each flask and filtered through a Millipore® filter, (BD, 0.6 μm). The filter was then dried at 90°C, cooled in a desiccator to room temperature, and weighed.

The percent change in dry cell weight concentration of the sample as compared to a control was calculated after 7, 12, and 14 days of exposure. The concentration of secondary or tertiary effluent at which the percent change in dry cell weight was 50% that of the control (EC_{50}) was then determined.

Daphnia Assay

Effluent samples were shipped to the Aquatic Toxicology Laboratory of EG&G, Bionomics Marine Research Laboratory at Wareham, Massachusetts, where they were stored at 4°C until tested. *Daphnia magna* (<24 hr old) used in the test were from laboratory stocks cultured at EG&G, Bionomics. Procedures used were based on protocols established by the EPA (3).

Two independent tests involving two different series of concentrations were generally performed through the study. A preliminary (range-finding) test was performed to define the narrower range of concentrations to be used in a subsequent definitive test. Mortality data derived from the definitive test were used to calculate a median lethal concentration (LC_{50}) and its 95% confidence limit utilizing least squares regression analysis. The LC_{50} is the calculated nominal concentration of the test compound in diluent water which produces 50% mortality in the test animal population at the stated times of exposure.

The static toxicity tests were conducted in 250-mℓ beakers which contained 150 mℓ of test solution. For each test concentration, the appropriate amount of the effluent was introduced into the required volume of diluent water to total 500 mℓ to 750 mℓ and mixed with a magnetic stirrer. This solution was then divided into three 150-mℓ aliquots in triplicate beakers to provide replicate exposure treatments. The remaining 50 mℓ were used for 0-hr dissolved oxygen (DO), specific conductance, alkalinity, total hardness, and pH determinations. Five daphnids were randomly assigned to each test vessel within 30 min after the effluent was added for a total of 15 daphnids per concentration. A control, consisting of the same dilution water and conditions but with no effluent, was established. All test vessels were maintained at constant temperature, and occasionally the test solutions had to be aerated. Dissolved oxygen, temperature, and pH were monitored through each test.

Fish Assay (Fathead Minnow and Bluegill)

Effluent samples were shipped to the Aquatic Toxicology Laboratory of EG&G, Bionomics Marine Research Laboratory at Wareham, Massachusetts, where they were stored at 4°C until tested. Fathead minnows (*Pimephales promelas*) were obtained from a commercial fish supplier in Missouri, and bluegills (*Lepomis macrochirus*) were obtained from a commercial fish supplier in Connecticut. Upon receipt at the Bionomics lab, fish were held until use (a minimum of 14 days) in a 1,700-ℓ raceway coated with epoxy paint. During this time period, all fish were fed a dry pelleted food daily, *ad libitum*, and ground liver weekly except during 48 hr prior to testing. There was less than a 2% mortality observed during this 2-day period.

Procedures used for the fish bioassay were based on protocols developed by the EPA (3). The toxicity test was conducted in 19.6-ℓ glass jars which contained 15 ℓ of test solution. The effluent was mixed with diluent water to provide the appropriate percentage concentrations. A control jar containing the same dilution water and maintained under the same conditions as test concentrations, but containing no secondary or tertiary effluent, was established. Test solution temperatures were controlled by a system designed to maintain test temperatures at 22 ± 1°C. Test solutions generally were not aerated. Ten fish were randomly distributed to each test jar within 3 hr after the test solutions were mixed. During the toxicity determination, the pH, temperature, and DO concentration of test solutions were measured at 0, 24, 48, 72, and 96 hr in the control, high, middle, and low test concentrations. The specific conductance, total hardness and alkalinity were measured in the control, high, middle, and low test concentrations at 0 hr. The concentrations tested and the corresponding mortality data derived from the toxicity test were used to establish 24-, 48-, 72- and 96-hour median lethal concentrations (LC_{50}) and 95% confidence intervals. The LC_{50} is defined as the concentration (nominal or measured) of the test compound in diluent water which causes 50% mortality in the test animal population at the stated exposure interval.

Determination of Mutagenicity

The effluent samples were shipped to MRC and stored at 4°C until tested. The samples were filter sterilized through 0.45-μm and 0.22-μm filters before application to the bioassay system. When streaked on nutrient agar, the samples exhibited bacterial contamination. The five strains of *Salmonella* (TA98, TA100, TA1535, TA1537, and TA1538) were obtained from Dr. Bruce Ames of the University of California. The strains are kept at -80°C and a scraping made each day to grow an overnight nutrient broth culture. The stock cultures are routinely checked for their genotypic characteristics and the presence of the plasmid.

For each experiment, the following solutions, listed in the order of addition, were added to a sterile 15 mm x 85 mm culture tube placed in a 45°C water bath:

- 2.00 mℓ of 0.6% agar (containing 0.05 mM histidine and 0.05 mM biotin)
- 0.10 mℓ of indicator organisms
- 0.50 mℓ of metabolic activation mixture
- 0.01 to 1.0 mℓ of the effluent sample

The metabolic activation mixture used for each experiment consisted of:

- 1.0 mℓ of S-9 rat liver fraction
- 0.2 mℓ of magnesium chloride (0.4 M) and potassium chloride (1.65 M)
- 0.10 mℓ of glucose-6-phosphate (1 M)
- 0.4 mℓ of nicotine adenine dinucleotide phosphate (4 μM)
- 5.0 mℓ of sodium phosphate (0.2 M, pH 7.4)
- 3.3 mℓ of water

The mixture in top agar with or without the metabolic activation mixture was vortexed and poured onto minimal agar plates. The plates consisted of the following, per milliliter:

15 g agar
20 g glucose
0.2 g of magnesium sulfate
2 g of citric acid monohydrate
10 g of potassium phosphate dibasic
3.5 g of sodium ammonium phosphate

After the top agar mixture solidified, the plates were inverted and incubated at 37°C for 48 hr. The number of revertant colonies was then counted with a 3M Colony Counter.

All samples were run in duplicate at five dosages, with and without metabolic activation. Positive and negative controls were run with all samples. The positive controls included 2-aminoanthracene, 2-nitrofluorene, and sodium nitrite.

The *E. coli* pol $A^{+/-}$ strains were stored at -80°C. Inoculums from the frozen stocks were grown overnight at 37°C with shaking in a liquid medium supplemented with 5 μg of thymine per mℓ (10).

For each test plate, 2 mℓ of top agar in a 16 mm x 85 mm tube was held at 45°C. To the 0.6% top agar, 0.1 mℓ overnight bacterial culture was added. This was mixed and poured onto plates. After the soft agar solidified, a sterile disc was placed in the center of the plate and the appropriate amount of filter-sterilized effluent was added to the disc. The plates were incubated at 37°C for 16 hr and then scored. The zone of toxicity or inhibition was measured on each plate. Several concentrations were tested for each sample.

Positive and negative controls were run along with all samples. The positive control was ethylmethanesulfonate. Larger zones of inhibition were observed with the DNA repair-deficient strain (p3478).

Determination of Cytotoxicity

The samples were filter sterilized through 0.45-µm and 0.22-µm filters prior to application to the test flask. All samples were kept at 4°C until tested. Samples were run at 5 to 7 concentration levels using approximately 300 to 500 cells that had been plated on the previous day. After incubation at 37°C for 6 days to 7 days, the media and sample were removed and the cells were fixed, stained, and counted with a Fisher counter. The data were entered into the laboratory computer to obtain the mean, standard deviation, and percent survival of each sample versus controls.

Positive and negative controls were run with all samples. The positive control was cadmium chloride. All samples were run in triplicate at 5 concentrations. A detailed procedure for this assay is given in Table 4.

TABLE 4. CHO-K1 CLONAL CYTOTOXICITY TEST

Cell line:	Chinese hamster ovary epithelial cells ATCC No. CCL 61
Medium:	F-12 GIBCO No. H-17 10.8 g/mℓ Sodium hydrogen carbonate 10% Fetal calf serum, virus, mycoplasma screened GIBCO No. 629
Incubation:	37°C, 5% CO_2, Saturated humidity
Samples:	6 Controls (blank) 5 to 7 Concentrations of test compound in triplicate 5 Concentrations of a positive toxic control in triplicate

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Test procedure

To stock CHO-K1, add 5 mℓ 0.25% trypsin at 37°C for 5 min.

Shake cells and add to centrifuge tube.

Add 5 mℓ media to flask, shake, and add to centrifuge tube.

Centrifuge 5 min at 1,200 G and pour off liquid, retaining cells.

Add 10 mℓ medium, shake, centrifuge 5 min, pour off medium.

Add 10 mℓ medium, shake.

Make hemocytometer count of trypsinized cells.

Dilute so that 5 mℓ media contain 300 to 500 cells.

Add 5 mℓ media and cells to T-25 flasks.

Incubate 12 hr to 18 hr to allow attachment using normal media.

Replace 5 mℓ of media and sample.

Incubate 6 days to 7 days total.

Fix with 10% formaldehyde/0.5% sodium chloride/4% methanol for 30 min.

Stain with crystal violet (0.04% for 15 min).

Count clonal colonies of remaining cells macroscopically using Fisher Count-All Model 600.

Score with respect to experimental vs. controls as percent survival.

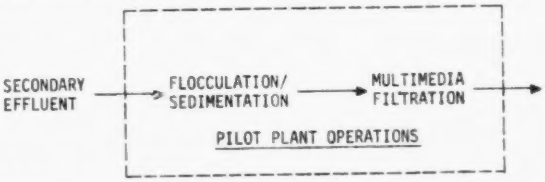
SECTION 8

ANALYTICAL AND BIOASSAY RESULTS

INTRODUCTION

Results of chemical analyses and bioassay of the wastewater streams sampled at each of the eight pilot plant sites are presented in the following subsections. All analytical and bioassay data acquired from each pilot plant site are presented together in a separate subsection for each site.

As discussed previously, individual unit processes were often operated in series during "candidate mode" studies when MRC conducted its sampling/analysis program. In general, MRC collected samples of the secondary effluent and samples after each unit operation in each candidate treatment system. For example, if the following unit processes were operated in series:



then samples of secondary effluent, flocculation/sedimentation effluent, and multimedia filter effluent were sampled and subsequently analyzed. Thus, the example sampling/analysis program would assess the performance of two tertiary process systems: flocculation/sedimentation, and multimedia filtration preceded by flocculation/sedimentation.

In this same manner, several tertiary process systems of the tertiary pilot plant were characterized at each textile mill location, simply by conducting the sampling/analysis program between unit processes in series. Tertiary process systems evaluated in the Phase II program fell into eight categories, as shown in Figure 1 (repeated here for reader convenience). Resulting chemical and bioassay data are classified in tables of the following subsections based on the tertiary process system type operated at each pilot plant location. Table 5 indicates which types of tertiary process systems were employed at each location. At the end of each subsection, chemical analysis/bioassay results are presented in five tables: results of organic

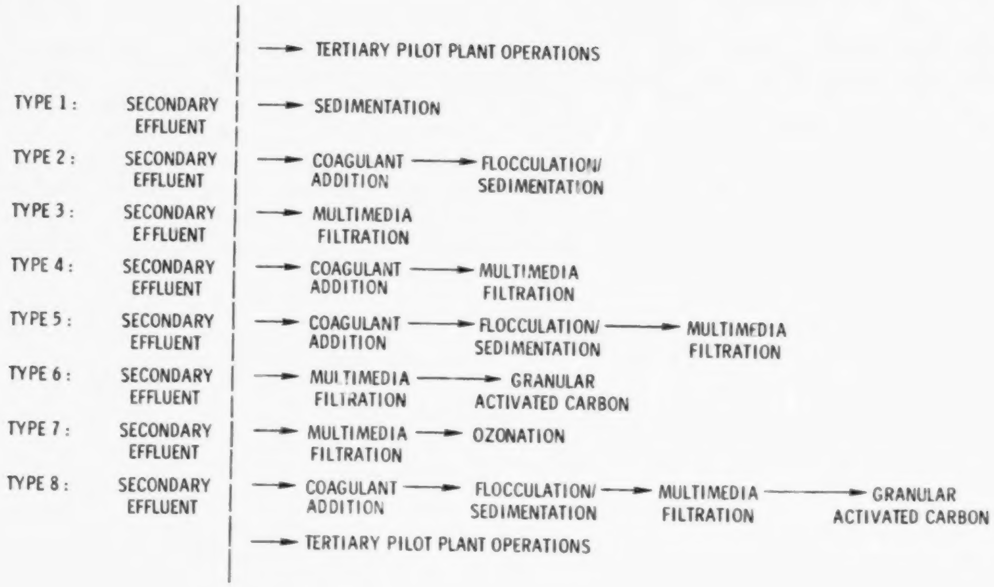

TERTIARY PILOT PLANT OPERATIONS
TYPE 1 : SECONDARY EFFLUENT → SEDIMENTATION
TYPE 2 : SECONDARY EFFLUENT → COAGULANT ADDITION → FLOCCULATION/ SEDIMENTATION
TYPE 3 : SECONDARY EFFLUENT → MULTIMEDIA FILTRATION
TYPE 4 : SECONDARY EFFLUENT → COAGULANT ADDITION → MULTIMEDIA FILTRATION
TYPE 5 : SECONDARY EFFLUENT → COAGULANT ADDITION → FLOCCULATION/ SEDIMENTATION → MULTIMEDIA FILTRATION
TYPE 6 : SECONDARY EFFLUENT → MULTIMEDIA FILTRATION → GRANULAR ACTIVATED CARBON
TYPE 7 : SECONDARY EFFLUENT → MULTIMEDIA FILTRATION → OZONATION
TYPE 8 : SECONDARY EFFLUENT → COAGULANT ADDITION → FLOCCULATION/ SEDIMENTATION → MULTIMEDIA FILTRATION → GRANULAR ACTIVATED CARBON
TERTIARY PILOT PLANT OPERATIONS

Figure 1. Tertiary treatment systems.

TABLE 5. TERTIARY TREATMENT SYSTEMS USED AT SPECIFIC PILOT PLANT SITES

	Type of tertiary treatment system studied							
Plant	1	2	3	4	5	6	7	8
A	-[a]	X			X			X
C		X			X			X
W	X		X			X	X	
S			X	X		X		
P		X	X		X	X		
N			X		X	X		
V			X	X		X	X	
T			X		X	X		

[a] Blanks indicate treatment technology not tested in "candidate" mode studies at this location.

toxic pollutant analyses, results of inorganic toxic pollutant analyses, results of other pollutant analyses, bioassay results, and effluent descriptions.

Tables indicating results of organic toxic pollutant analyses present concentrations of only those organic compounds on the toxic pollutant list (see the appendix) that were found above analytical detection limits (see Table 6) in the wastewater sample. In other words, all organic compounds found on the toxic pollutant list were analyzed by the chemical analytical technique formerly discussed (see Section 6), but only those observed in the wastewater samples are listed in the organic toxic pollutant tables.

The same tables present concentrations of pollutants found in the textile plant intake water at the time of Phase II sampling, concentrations of pollutants found in the secondary effluent during the Phase I sampling/analysis program (4), and concentrations of pollutants found in the secondary and tertiary process system effluents during the Phase II sampling/analysis program.

In addition, some pollutant removal efficiencies of the tertiary process systems were calculated. Since results of toxic pollutant analyses only indicate general concentrations, the presented removal efficiency data must be treated with extreme care. These data are only useful to indicate general trends. Removal efficiencies were calculated only in cases where the Phase II secondary effluent or tertiary process series effluent exceeded 10 $\mu g/\ell$, since low level contamination of the samples for some toxic pollutant species often occur in the analyses procedure. The removal efficiencies presented were calculated as follows:

$$\frac{A - B}{A} \times 100 = \%\ \text{removal}$$

where A = concentration of pollutant species present in the Phase II secondary effluent.

B = concentration of pollutant species present in the Phase II tertiary effluent.

When A or B was less than the detection limit, the detection limit was used in the calculation.

Tables indicating results of inorganic toxic pollutant analyses and other pollutant analyses are organized in much the same way as tables presenting results of organic toxic pollutant analyses.

Tables of bioassay results present results of the bioassay procedures described in Section 7 for secondary and tertiary effluents only, since bioassays were not conducted on intake water in the Phase II program. Since the specific bioassay parameter measured

TABLE 6. MINIMUM DETERMINABLE CONCENTRATIONS FOR ORGANIC TOXIC POLLUTANTS

Compound	Concentration, µg/liter
Acids:	
2-Chlorophenol	0.09
Phenol	0.07
2,4-Dichlorophenol	0.1
2-Nitrophenol	0.4
p-Chloro-*m*-cresol	0.1
2,4,6-Trichlorophenol	0.2
2,4-Dimethylphenol	0.1
2,4-Dinitrophenol	2.0
4,6-Dinitro-*o*-cresol	40.0
4-Nitrophenol	0.9
Pentachlorophenol	0.4
Volatiles:	
Chloromethane	0.2
Dichlorodifluoromethane	0.2
Bromomethane	0.2
Vinyl chloride	0.4
Chloroethane	0.5
Methylene chloride	0.4
Trichlorofluoromethane	2.0
1,1-Dichloroethylene	2.0
1,1-Dichloroethane	3.0
Trans-1,2-dichloroethylene	2.0
Chloroform	5.0
1,2-Dichloroethane	2.0
1,1,1-Trichloroethane	2.0
Carbon tetrachloride	4.0
Bromodichloromethane	0.9
Bis(chloromethyl) ether	1.0
1,2-Dichloropropane	0.7
Trans-1,3-dichloropropene	0.4
Trichloroethylene	0.5
Dibromochloromethane	0.3
Cis-1,3-dichloropropene	0.5
1,1,2-Trichloroethane	0.7
Benzene	0.2
2-Chloroethyl vinyl ether	1.0
Bromoform	0.6
Tetrachloroethylene	0.9
1,1,2,2-Tetrachloroethane	0.6
Toluene	0.1
Chlorobenzene	0.2
Ethylbenzene	0.2
Direct Injectables:	
Acrolein	200
Acrylonitrile	100
Base/Neutrals:	
1,3-Dichlorobenzene	0.02
1,4-Dichlorobenzene	0.04
Hexachloroethane	0.1
1,2-Dichlorobenzene	0.05
Bis(2-chloroisopropyl) ether	0.06
Hexachlorobutadiene	0.08
1,2,4-Trichlorobenzene	0.09
Naphthalene	0.007
Bis(2-chloroethyl) ether	0.07
Hexachlorocyclopentadiene	0.2
Nitrobenzene	0.08
Bis(2-chloroethoxy) methane	0.06
2-Chloronaphthalene	0.02
Acenaphthylene	0.02
Acenaphthene	0.04
Isophorone	0.06
Fluorene	0.02
2,6-Dinitrotoluene	0.2
1,2-Diphenylhydrazine	0.02
2,4-Dinitrotoluene	0.02
N-nitrosodiphenylamine	0.07
Hexachlorobenzene	0.05
4-Bromophenyl phenyl ether	0.1
Phenanthrene	0.01
Anthracene	0.01
Dimethyl phthalate	0.03
Diethyl phthalate	0.03
Fluoranthene	0.02
Pyrene	0.01
Di-n-butyl phthalate	0.02
Benzidine	0.02
Butyl benzyl phthalate	0.03
Chrysene	0.02
Bis(2-ethylhexyl) phthalate	0.04
Benz(a)anthracene	0.02
Benzo(b)fluoranthene	0.02
Benzo(k)fluoranthene	0.02
Benzo(a)pyrene	0.02
Indeno(1,2,3-cd)pyrene	0.02
Dibenzo(a,h)anthracene	0.02
Benzo(g,h,i)perylene	0.01
N-Nitrosodimethylamine	0.8
N-nitrosodi-n-propylamine	0.2
4-Chlorophenyl phenyl ether	0.03
3,3'-Dichlorobenzidine	1.0
Di-n-octyl phthalate	0.9
Pesticides and PCB's	1.0

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[a] Based on the lowest quantifiable area obtained from the GC/MS.

varied from test to test, a column indicating the measured parameter is included in the bioassay results tables. Explanation of these parameters can be found in Section 7. Where EC_{50}'s or LC_{50}'s were the measured parameters, 95% confidence intervals are also stated.

A table of effluent descriptions is also included with each pilot plant assessment. Results presented in these tables generated concurrently by individuals conducting static acute toxicity tests.

The chemical analysis/bioassay data discussions include overviews of background material, sampling/analysis deviations from protocols described in earlier discussions, and signficiant trends specific to the results from each of the eight pilot plant studies. A more complete evaluation of the data is provided in Section 9.

PLANT DATA

Plant A

Textile plant A generated wastewater from wool finishing operations. Existing treatment facilities included screens, equalization, aeration, secondary clarification, and chlorination; sludge dewatering techniques included dissolved air flotation and vacuum filtration. Phase I screening studies (4) indicated that plant A wastewater treated by the existing treatment system was 1 of the more toxic effluents of the 22 secondary effluents actually evaluated in the program. Therefore, plant A was included as a site for tertiary pilot plant studies and MRC sampling/analysis.

After tertiary process screening studies were conducted by Engineering Science, Inc., it was decided that tertiary treatment types 2, 5, and 8 held the best promise for improving secondary effluent quality to meet suggested BATEA guidelines; they were thus selected for candidate mode studies. Alum and lime were used as coagulants in each of the treatment systems. A flow diagram of the candidate mode configuration employed is shown in Figure 10. Sampling of the candidate mode operation was conducted by MRC, and results of subsequent chemical analyses and bioassay tests are presented in Tables 7 through 11.

In evaluating the data, it is apparent that type 2 treatment (flocculation/sedimentation) removed significant amounts of most of the organic toxic pollutants found in the secondary effluent, except for bis(2-ethylhexyl) phthalate. Subsequent multimedia filtration (type 5) and granular activated carbon treatment (type 8) generally removed additional amounts, but type 5 treatment appeared to add trace amounts of some organic pollutants such as total phenol, benzo(a)pyrene, specific phenols, and

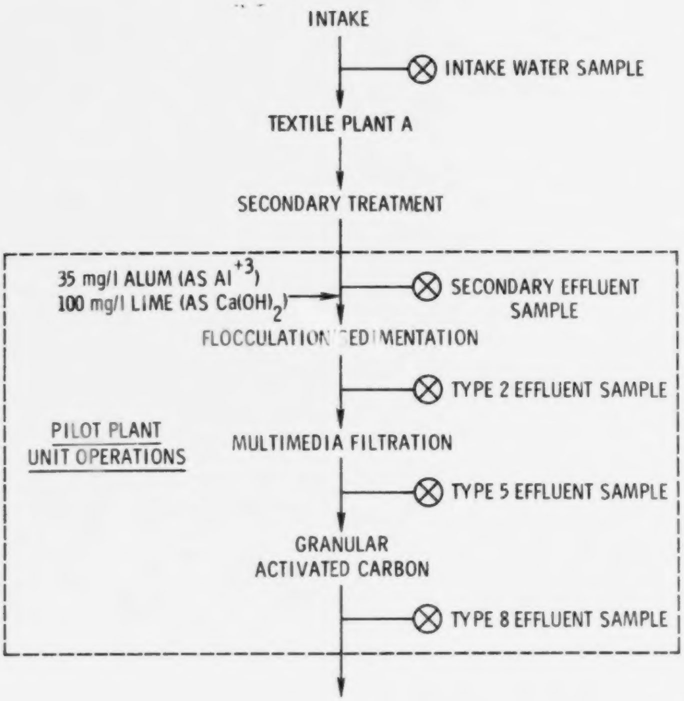
INTAKE
INTAKE WATER SAMPLE
TEXTILE PLANT A
SECONDARY TREATMENT
35 mg/l ALUM (AS Al+3)
100 mg/l LIME (AS Ca(OH)2)
SECONDARY EFFLUENT SAMPLE
FLOCCULATION/SEDIMENTATION
TYPE 2 EFFLUENT SAMPLE
PILOT PLANT UNIT OPERATIONS
MULTIMEDIA FILTRATION
TYPE 5 EFFLUENT SAMPLE
GRANULAR ACTIVATED CARBON
TYPE 8 EFFLUENT SAMPLE

Figure 10. Candidate wastewater treatment system studied at plant A.

dichlorobenzenes. A possible source of these contaminants was leaching from the filter media used in type 5 treatment. Contaminants may have been adsorbed onto the filter media during a former pilot plant study, or they may have been present in the virgin filter media.

Except for chromium, copper, cyanide, and zinc, inorganic toxic pollutants were added to the secondary effluent by the tertiary process series. Most of the chromium (72%) was removed by type 2 treatment (flocculation/sedimentation), with subsequent filtration (type 5) and activated carbon treatment (type 8) doing little to remove additional chromium. Cyanide was removed by type 2 and type 8 treatment, and copper was removed to a slight degree (6% to 11%) by all three types of tertiary treatment. As expected, aluminum and calcium concentrations increased through tertiary treatment because alum and lime were used as coagulants. Phosphorus was removed (more than 75%) by all three treatment systems. This is expected since alum and lime coagulation are classical methods of phosphorus removal.

In evaluating bioassay results, it is apparent that the secondary effluent was as acutely toxic during Phase II sampling/analysis as it was during Phase I sampling/analysis. Slight improvement in toxicity to freshwater algae was observed when type 2 treatment (flocculation/sedimentation) was employed. Subsequent filtration (type 5) and activated carbon treatment (type 8) did little to improve toxicity to freshwater algae.

All three types of tertiary treatment were effective in removing toxicity to *Daphnia*. Type 8 treatment (flocculation/sedimentation followed by filtration and activated carbon treatment) improved the secondary effluent with regard to acute toxicity to *Daphnia* to the point that no acute toxicity was observed. Type 2 and type 5 treatment systems added something to the wastewater (possibly aluminum or calcium used in coagulation), which was toxic to bluegills. However, subsequent treatment of the wastewater by granular activated carbon improved the toxic effect to the point where no toxicity was observed.

TABLE 7. PLANT A ORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

		Secondary effluent		Tertiary effluent					
Pollutant	Intake	Phase I	Phase II	Type 2		Type 5		Type 8	
Bis(2-ethylhexyl) phthalate	5.4	6[b]	32	44	(-38)[a]	14	(56)	4.7	(85)
Pyrene	1.2	-							
Heptachlor		1.6	1.4						
1,2-Dichlorobenzene		1	20		(100)	5.4	(73)		(100)
1,2,4-Trichlorobenzene		46	1,600	150	(91)	94	(94)		(100)
α-BHC			5.8			1.9			
4,4'-DDT			2.1						
Toluene		8.4	31	14	(55)	12	(31)		(100)
Ethylbenzene			5						
Phenol				3		3		1.5	
Benzo(a)pyrene						0.8			
N-nitrosodiphenylamine						0.4			
2,4-Dimethylphenol						0.9			
Pentachlorophenol						10			
1,4-Dichlorobenzene		0.05							
Phenol (total)	12	65	60	47	(22)	55	(8)	17	(72)

[a] Minus percent removals indicate an increase in the concentration of the specific pollutant.

[b] Blanks indicate concentration below detection limit (see Table 6).

TABLE 8. PLANT A INORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, $\mu g/\ell$; percent removal in parentheses)

		Secondary Effluent		Tertiary effluent					
Pollutant	Intake	Phase I	Phase II	Type 2		Type 5		Type 8	
Antimony	<10	30	<10	<10		<10		24[a]	(-140)[b]
Arsenic	72	<5	60	62	(-3)	103	(-72)	<1	(>98)
Beryllium	0.2	NA[c]	<0.04	<0.04		1.2	(-2,900)	5.4	(-130,000)
Cadmium	7.5[a]	<0.5	<2	<2		97	(-4,800)	5.2[a]	(-62)
Chromium	<4	180	110	31	(72)	34	(69)	19[a]	(83)
Copper	<4	27	20	13[a]	(35)	110	(-4,500)	47	(-140)
Cyanide	<4	15	10	<4	(>60)	10	(0)	<4	(>60)
Lead	<22	<1	<22	<22		79[a]	(-260)	<22	
Mercury	NA	<0.5	NA	NA		NA		NA	
Nickel	<36	140	<36	<36		<36		<36	
Selenium	NA	<5	NA	NA		NA		NA	
Silver	<5	<5	<5	<5		<5		<5	
Thallium	NA	<5	NA	NA		NA		NA	
Zinc	56	6,400	6,400	5,700	(11)	5,900	(8)	6,000	(6)

[a] Semiquantitative region; value not within 95% confidence limits.

[b] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[c] Not analyzed.

TABLE 9. PLANT A OTHER POLLUTANTS DETECTED
(Concentration, µg/ℓ; percent removal in parentheses

			Tertiary effluent					
		Secondary	Type 2		Type 5		Type 8	
Pollutant	Intake	effluent						
Aluminum	<12	230	1,600	(-600)[a]	520	(-130)	100	(57)
Barium	<0.2	20	18	(10)	18	(10)	50	(-150)
Boron	50	270	270	(0)	300	(-11)	320	(-19)
Calcium	16,000	37,000	70,000	(-89)	70,000	(-89)	70,000	(-89)
Cobalt	7.7[b]	16[b]	17[b]	(-6)	110	(-590)	18[b]	(-13)
Iron	175	2,000	2,800	(-40)	2,700	(-35)	1,700	(15)
Magnesium	3,000	5,000	4,900	(2)	5,300	(-6)	5,800	(-16)
Manganese	49	92	100	(-9)	200	(-120)	120	(-30)
Molybdenum	<10	<10	<10		<10		<10	
Phosphorus	490[b]	280	<70	(>75)	<70	(>75)	<70	(>75)
Sodium	8,000	270,000	270,000	(0)	280,000	(-4)	265,000	(2)
Silicon	480	1,400	1,300	(7)	1,300	(7)	1,400	(0)
Strontium	90	220	240	(-9)	260	(-18)	370	(-68)
Tin	<15	<15	20[b]	(-33)	17[b]	(-13)	<15	
Titanium	<1.0	3.2[b]	2.0[b]	(38)	<1.0	(>69)	<1.0	(>69)
Vanadium	17	40	53	(33)	42	(-5)	33	(18)

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[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Semiquantitative region; value not within 95% confidence limits.

TABLE 10. PLANT A BIOASSAY RESULTS

Test species	Parameters	Secondary effluent		Tertiary effluent		
		Phase I	Phase II	Type 2	Type 5	Type 8
Freshwater algae - *S. capricornutum*	EC_{50} - 7 day, % effluent	NM[a]	15 (4.2-52)[b]	73 (46-100)	38 (12-100)	42 (26-68)
	EC_{50} - 12 day, % effluent	NM	18 (5.9-52)	>32<56	36 (12-100)	26 (11-61)
	EC_{50} - 14 day, % effluent	-[c]	22 (5.6-87)	30 (6.9-100)	30 (13-63)	31 (16-61)
Daphnia - *D. magna*	LC_{50} - 24 hr, % effluent	NM	>100	<100	41 (31-50)	>100
	LC_{50} - 48 hr, % effluent	9[d]	25 (17-32)	56 (49-62)	57 (50-63)	>100
Bluegill - *L. macrochirus*	LC_{50} - 24 hr, % effluent	NM	-[e]	71 (64-79)	74 (61->100)	>100
	LC_{50} - 48 hr, % effluent	NM	-[e]	53 (41-69)	44 (37-58)	>100
	LC_{50} - 72 hr, % effluent	NM	73 (64-85)	42 (37-47)	40 (35-46)	>100
	LC_{50} - 96 hr, % effluent	NM	54 (36-88)	38 (33-43)	35 (32-40)	>100
Fathead minnow - *P. promelas*	LC_{50} - 96 hr, % effluent	19	NM	NM	NM	NM
S. typhimurium - strains TA98, TA100, TA1535, TA1537, and TA1538	Response to Ames test for mutagenicity - (-) or (+)	(-)	(-)[f]	NM	NM	NM

[a] Not measured.

[b] 95% confidence interval.

[c] 20% secondary effluent failed to support the growth of *S. capricornutum*.

[d] EC_{50} - 48 hr determined with *Daphnia pulex*.

[e] Insufficient data to calculate LC_{50}.

[f] This sample was also concentrated to 1000X; an increase in revertants were observed using the concentrate and TA98; however, no dose response was observed.

TABLE 11. PLANT A EFFLUENT DESCRIPTIONS

Parameter	Secondary effluent		Tertiary effluent		
	[redacted]	Phase II	Type 2	Type 5	Type 8
Physical description	Gray with considerable amount of fine particulate; chlorinated	[redacted] particulate matter	Light purple [redacted]	Light purple with [redacted] matter	Clear [redacted]
pH	6.2	6.6	6.1	6.6	6.2
Salinity, g/ℓ	NM[a]	0.05	0.02	0.05	0.05
Specific conductivity, μmhos/cm²	NM	850	900	850	900

[a] Not measured.

Plant C

Textile plant C generated wastewater from woven fabric finishing operations. Existing treatment facilities included screens, pH adjustment, two-stage aeration, and secondary clarification. Sanitary wastewater was chlorinated prior to discharge into the treatment plant influent. Phase I screening studies (4) indicated that plant C wastewater treated by the existing treatment facilities was 1 of the more toxic effluents of the 22 secondary effluents actually evaluated in the program. Therefore, plant C was included as a site for Phase II tertiary pilot plant studies and MRC sampling/analysis.

After tertiary process screening studies were conducted by Engineering Science, Inc., it was decided that tertiary treatment types 2, 5, and 8 held the most promise of improving secondary effluent quality to meet suggested BATEA guidelines, and they were selected for candidate mode studies. Alum was used as a coagulant in all three systems. A flow diagram of the employed candidate mode configuration is shown in Figure 11. Sampling of the candidate mode operation was conducted by MRC, and results of subsequent chemical analyses and bioassay tests are presented in Tables 12 through 16.

Low levels of toluene, total phenol, and other organic toxic pollutants were removed by type 2 (flocculation/sedimentation) treatment; however, levels of bis(2-ethylhexyl) phthalate and 1,2-dichlorobenzene increased. Bis(2-ethylhexyl) phthalate and 1,2-dichlorobenzene were subsequently removed by filtration (type 5), while toluene and total phenol levels were further reduced. As in plant A, the level of pentachlorophenol increased by type 5 treatment but was reduced by type 8 treatment (subsequent granular activated carbon treatment). Type 8 treatment also removed remaining levels of total phenol, but bis(2-ethylhexyl) phthalate levels increased.

Except for copper, silver, and zinc, levels of inorganic toxic pollutants increased or remained the same after all three types of treatment. Most of the copper appearing in the secondary effluent was removed by type 2 treatment, and type 8 treatment

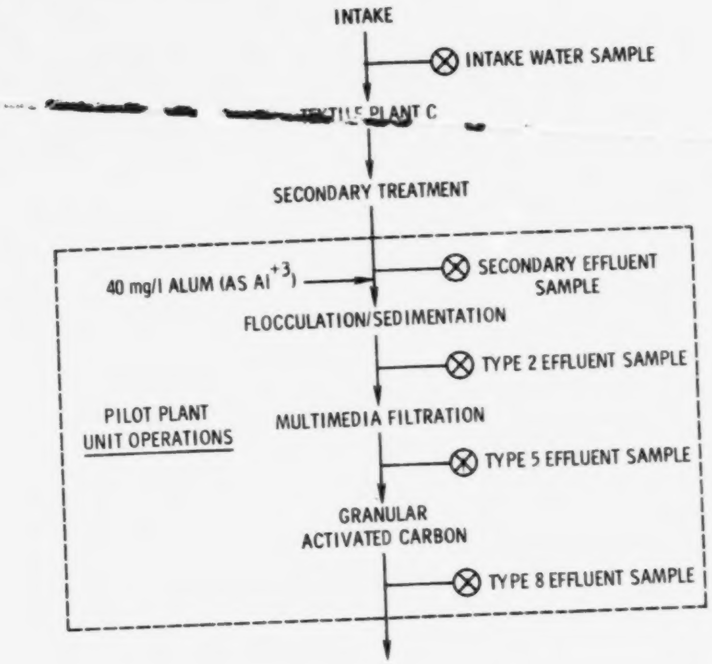
INTAKE
INTAKE WATER SAMPLE
TEXTILE PLANT C
SECONDARY TREATMENT
40 mg/l ALUM (AS Al+3)
SECONDARY EFFLUENT SAMPLE
FLOCCULATION/SEDIMENTATION
TYPE 2 EFFLUENT SAMPLE
PILOT PLANT UNIT OPERATIONS
MULTIMEDIA FILTRATION
TYPE 5 EFFLUENT SAMPLE
GRANULAR ACTIVATED CARBON
TYPE 8 EFFLUENT SAMPLE

Figure 11. Candidate wastewater treatment system studied at plant C.

removed zinc. As expected since alum was used as a coagulant, aluminum concentrations increased through tertiary treatment.

In evaluating bioassay data, it is apparent that flocculation/ sedimentation with alum was detrimental to freshwater algae and *Daphnia*. The wastewater became more toxic to these species after type 2 treatment (flocculation/sedimentation) and never recovered to the original toxicity levels after subsequent filtration (type 5) and granular activated carbon treatment (type 8). No toxicity was observed in any of the effluents when fathead minnows or bluegills were used as the test species. Microbiological bioassay work provided no indication of effluent improvement or degradation by the treatment types.

TABLE 12. PLANT C ORGANIC TOXIC POLLUTANTS DETECTED (Concentration, μg/ℓ; percent removal in parentheses)

		Secondary effluent		Tertiary effluent					
Pollutant	Intake	Phase I	Phase II	Type 2		Type 5		Type 8	
D[illegible] butyl phthalate	1.9	-[a]	0.6	0.6		0.6		0.4	
Bis(2-ethylhexyl) phthalate	6.6	3.0	7.6	33	(-330)[b]	5.3	(30)	11	(-45)
2-Chlorophenol	0.2								
Anthracene		4.4	0.05	0.1		0.03		0.01	
Pentachlorophenol						12	(-79)		
Phenol	0.9		0.4						
Toluene			15	1.0	(93)		(>99)		(>99)
Dibromochloromethane			0.6						
1,2-Dichlorobenzene		0.3		13	(-260,000)	5.8			
Ethylbenzene				1.3					
Acenaphthene		0.5							
1,2,4-Trichlorobenzene		10							
Methylene chloride[c]	35		160	210	(-31)	110	(31)		(100)
Phenol (total)		88	23	16	(30)	19	(17)		(>91)

[a] Blanks indicate concentration below detection limit (see Table 6).

[b] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[c] Methylene chloride may originate from analysis contamination.

TABLE 13. PLANT C INORGANIC TOXIC POLLUTANTS DETECTED (Concentration, μg/ℓ; percent removal in parentheses)

		Secondary effluent		Tertiary effluent					
Pollutant	Intake	Phase I	Phase II	Type 2		Type 5		Type 8	
Antimony	<10	4	96	120	(-25)[a]	140	(-46)	120	(-25)
Arsenic	<1	<5	1.6	<1		<1		<1	
Beryllium	30	<0.1	1.5	2.2		1.2		2.7	
Cadmium	29	6	<2	2.9[b]		2.7[b]		9.8[b]	
Chromium	<4	31	5.5[b]	17[b]	(-210)	14[b]	(-150)	15[b]	(-170)
Copper	65	20	57	11[b]	(81)	25	(56)	35	(39)
Cyanide	<4	13	<4	<4		<4		<4	
Lead	<22	120	27[b]	66[b]	(-140)	64[b]	(-140)	64[b]	(-140)
Mercury	NA[c]	0.7	NA	NA		NA		NA	
Nickel	<36	140	<36	<36		<36		<36	
Selenium	NA	<5	NA	NA		NA		NA	
Silver	54	<5	80	72	(10)	77	(4)	91	(14)
Thallium	NA	<5	NA	NA		NA		NA	
Zinc	53	120	160	190	(-19)	230	(-44)	83	(48)

[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Semiquantitative region; value not within 95% confidence limits.

[c] Not analyzed.

TABLE 14. PLANT C OTHER POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

			Tertiary effluent					
		Secondary	Type 2		Type 5		Type 8	
Pollutant	Intake	effluent						
Aluminum	<12	98	13,000	(-13,000)[a]	11,000	(-10,000)	9,200	(-9,300)
Barium	81	72	70	(3)	71	(1)	81	(-13)
Boron	<1	54	58	(-7)	57	(-6)	29	(46)
Calcium	3,300	5,200	5,700	(-10)	5,600	(-8)	5,600	(-8)
Cobalt	29[b]	<6	<6		<6		<6	
Iron	110	230	930	(-300)	750	(-230)	310	(-35)
Magnesium	720	3,700	3,700	(0)	3,700	(0)	3,700	(0)
Manganese	33	17	24	(-41)	24	(-41)	28	(-105)
Molybdenum	<10	<10	20[b]	(-100)	22[b]	(-120)	19[b]	(-90)
Phosphorus	120[b]	2,700	2,300	(15)	2,000	(26)	1,900	(30)
Silicon	7,500	15,000	15,000	(0)	15,000	(0)	14,000	(7)
Strontium	49	67	68	(-1)	71	(-6)	74	(-10)
Tin	<15	69[b]	62[b]	(10)	66[b]	(4)	56[b]	(19)
Titanium	<1	2[b]	14	(-600)	11	(-450)	11	(-450)
Vanadium	<2	410	560	(-37)	520	(-27)	180	(-56)

[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Semiquantitative region; value not within 95% confidence limits.

TABLE 15. PLANT C BIOASSAY RESULTS

Test species	Parameters	Secondary effluent		Tertiary effluent		
		Phase I	Phase II	Type 2	Type 5	Type 8
Freshwater algae - *S. capricornutum*	EC_{50} - 7 day, % effluent	NM[a]	63 (47-85)[b]	7.7 (2.3-25)	12 (1.2-100)	17 (0.3-100)
	EC_{50} - 12 day, % effluent	NM	56 (40-78)	5.6 (2.3-13)	6.5 (2.5-17)	7.4 (1.9-30)
	EC_{50} - 14 day, % effluent	-[c]	49 (27-89)	5.2 (1.3-20)	5.3 (2.2-13)	4.5 (2.2-9.4)
Daphnia *D. magna*	LC_{50} - 24 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 48 hr, % effluent	41[d]	>100	79	85	89
Bluegill - *L. machrochirus*	LC_{50} - 24 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 48 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 72 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 96 hr, % effluent	NM	>100	>100	>100	>100
Fathead minnow - *P. promelas*	LC_{50} - 24 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 48 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 72 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 96 hr, % effluent	47	>100	>100	>100	>100
S. typhimurium - strains TA98, TA100, TA1535, TA1537, and TA1538	Response to Ames test for mutagenicity - (-) or (+)	(-)	(-)[e]	(-)	NM	(-)
E. coli - strains W3110 and p3478	Response to pol A test for mutagenicity-zone of inhibition, mm	0	0	0	NM	0
Chinese hamster ovary cells	Response to CHO-K1 test for acute cytotoxicity-EC_{50}	-[f]	>100	>100	NM	>100

[a] Not measured.

[b] 95% confidence interval.

[c] 20% secondary effluent was highly stimulatory to the growth of *S. capricornutum*.

[d] EC_{50} - 48 hr determined using *Daphnia pulex*.

[e] Increase in number of revertants over background was observed; however, the results were not twofold, nor was there a dose response.

[f] EC_{50} not determinable; cytotoxicity procedure employing rabbit alveolar macrophage (RAM) used in Phase I.

TABLE 16. PLANT C EFFLUENT DESCRIPTIONS

Parameter	Secondary effluent		Tertiary effluent		
	Phase I	Phase II	Type 2	Type 5	Type 8
Physical description	Clear, blue-black with moderate amount of particulate; unchlorinated	Orange-brown with particulate matter	Light orange- with particulate matter	Light orange-brown with particulate matter	Cloudy white liquid
pH	10.2	8.3	6.9	7.0	7.1
Salinity, g/ℓ	NM[a]	3	3	3	2.5
Specific conductivity, umhos/cm^a	NM	3,800	4,000	3,900	3,900

[a] Not measured.

Plant W

Textile plant W generated wastewater from wool scouring operations. Existing treatment facilities included a grit chamber, oxidation ditch, secondary clarification, and chlorination, while waste sludge was treated on sludge drying beds. Phase I screening studies (4) indicated that plant W wastewater treated by the existing treatment facilities was 1 of the more toxic effluents of the 22 secondary effluents actually evaluated in the program. Therefore, plant W was included as a site for Phase II tertiary pilot plant studies and MRC sampling/analysis.

After tertiary process screening studies were conducted by Engineering Science, Inc., it was apparent that further clarification of the secondary effluent was necessary before subsequent tertiary process system studies could be made. Therefore, the reactor/clarifier contained in the tertiary pilot plant trailer was operated as an additional secondary clarifier (i.e., no coagulant addition) before the tertiary processes were evaluated. The MRC sampling/analysis program included sampling and analysis of the secondary effluent before and after the additional clarification. In tables of analytical/bioassay results which follow, the waste stream after additional clarification is referred to as type 1 treatment effluent; it is referred to as secondary effluent as it is generated by the existing facilities.

After the screening studies, it was determined that tertiary treatment types 3, 6, and 7 after additional clarification (type 1) held the most promise of improving secondary effluent quality to meet suggested BATEA guidelines and were thus selected for candidate mode studies. Figure 12 is a flow diagram of the candidate mode configuration used. Sampling of the candidate mode operation was conducted by MRC, and results of subsequent analysis and bioassay work are presented in Tables 17 through 21. Bis(2-ethylhexyl) phthalate and total phenol were the only organic toxic pollutants found in plant W secondary effluent at levels greater than 10 μg/ℓ. Type 1 treatment (additional clarification), type 3 treatment (subsequent filtration), and type 6 treatment

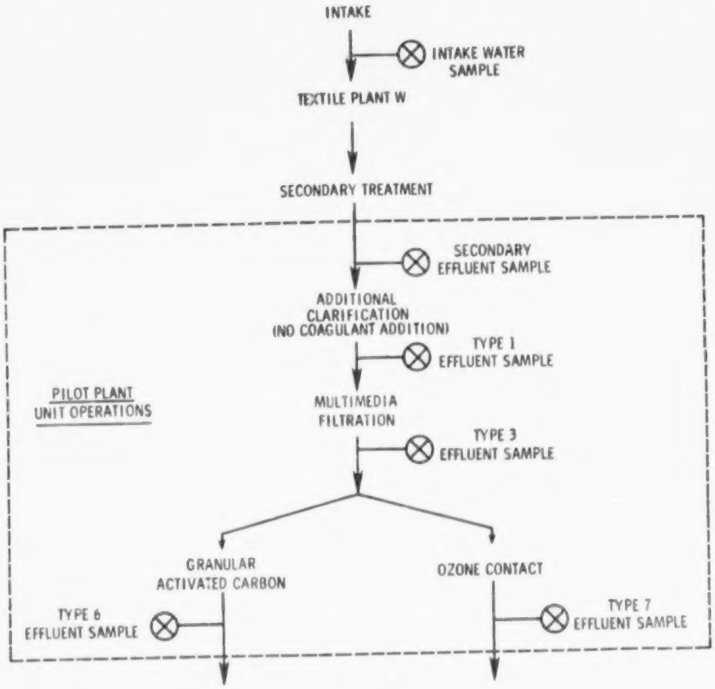
INTAKE
INTAKE WATER SAMPLE
TEXTILE PLANT W
SECONDARY TREATMENT
SECONDARY EFFLUENT SAMPLE
ADDITIONAL CLARIFICATION (NO COAGULANT ADDITION)
TYPE 1 EFFLUENT SAMPLE
PILOT PLANT UNIT OPERATIONS
MULTIMEDIA FILTRATION
TYPE 3 EFFLUENT SAMPLE
GRANULAR ACTIVATED CARBON
OZONE CONTACT
TYPE 6 EFFLUENT SAMPLE
TYPE 7 EFFLUENT SAMPLE

Figure 12. Candidate wastewater treatment system studied at plant W.

(subsequent activated carbon) removed some bis(2-ethylhexyl) phthalate, but this pollutant was added by type 7 treatment (ozonation following filtration and clarification). This observed addition of organic toxic pollutants by type 7 treatment can be attributed to leaching from the ozone contactor materials of construction. Total phenol was slightly removed (19%) by ozonation (type 7).

Antimony, cadmium, copper, nickel, silver, lead, and zinc were removed by additional clarification (type 1) alone. These same metals, except for lead, were added by type 7 treatment (ozonation preceded by additional clarification and filtration); they may have been added by decay of electrode plates in the ozone generator. Cyanide was removed by activated carbon and ozonation (types 6 and 7), while lead and zinc were removed by all four treatment types.

Type 1 treatment (additional clarification) removed significant amounts of aluminum, barium, cobalt, iron, silicon, titanium, and vanadium. Subsequent tertiary treatments (types 3 and 6) removed additional amounts of most of the metals, while ozonation (type 7) added aluminum, boron, cobalt, manganese, and vanadium.

In evaluating bioassay results, it is apparent that the secondary effluent was toxic to all species used in static acute toxicity tests. None of the treatment types significantly improved the toxicity of the wastewater. Microbiological bioassay work provided no indication of effluent improvement or degradation by the treatment types.

TABLE 17. PLANT W ORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent		Tertiary effluent							
		Phase I	Phase II	Type 2		Type 3		Type 6		Type 7	
Bis(2-ethylhexyl) phthalate	15	19	42	23	(45)	14	(67)	26	(67)	106	(-150)[a]
Anthracene	0.9	–[b]	1.5	0.4		0.2		0.1		0.4	
Benzo(a)anthracene	0.1		1.5								
Fluoranthene	0.7		1.1	0.4		0.2				0.1	
Benzo(a)pyrene	0.7		1.2			0.2					
Pyrene	0.5		0.8	0.2		0.3				0.1	
Benzo(k)fluoranthene	0.4		0.8			0.1					
Di-n-butyl phthalate								1.1			
Toluene		1.7	1.4	9.5				3.1		1.2	
Ethylbenzene				3.0				1.3			
Isophorone						3.1					
Methylene chloride[c]				2.2		4.8		1.8		61	(-15,000)
Phenol (total)	6	232	16	49	(-67)	17	(-6)	17	(-6)	13	(19)

[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Blanks indicate concentration below detection limit (see Table 6).

[c] Methylene chloride may originate from analysis contamination.

TABLE 18. PLANT W INORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent		Tertiary effluent							
		Phase I	Phase II	Type 1		Type 3		Type 6		Type 7	
Antimony	<10	<0.5	540	<200	(>63)	<200	(>63)	<200	(>63)	1,200	(-120)[a]
Arsenic	<1.0	4	38	39	(-3)	83	(-120)	42	(-10)	43	(-13)
Beryllium	<5	<0.1	<2	<2		<2		<2		<4	
Cadmium	<2	13	130	<40	(>69)	<40	(>69)	<40	(>69)	250	(-92)
Chromium	<4	3	<80	<80		<80		<80		<200	
Copper	<4	2	320	110	(66)	120	(63)	<80	(>75)	590	(-84)
Cyanide	<4	20	200	240	(-20)	260	(-30)	40	(80)	<4	(>98)
Lead	<20	57	3,500	<400	(>86)	<400	(>86)	<400	(>86)	<900	(>74)
Mercury	NA[b]	0.5	NA	NA		NA		NA		NA	
Nickel	<40	60	2,000	<700	(>65)	<700	(>65)	<700	(>65)	5,000	(-150)
Selenium	NA	<5	NA	NA		NA		NA		NA	
Silver	6	95	500	<100	(>80)	<100	(>80)	<100	(>80)	1,300	(-160)
Thallium	NA	<5	NA	NA		NA		NA		NA	
Zinc	17	90	1,500	190	(87)	400	(73)	120	(92)	460	(69)

[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Not analyzed.

TABLE 19. PLANT W OTHER POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent	Tertiary effluent							
			Type 1		Type 3		Type 6		Type 7	
Aluminum	22	8,400	4,700	(44)	3,100	(63)	3,100	(63)	7,000	(17)
Barium	110	290	120	(59)	110	(62)	50	(83)	120	(59)
Boron	<1	490	640	(-33)[a]	600	(-22)	630	(-29)	1,000	(-104)
Calcium	16,000	31,000	31,000	(0)	33,000	(6)	22,000	(29)	30,000	(3)
Cobalt	5	170	<40	(>76)	60	(65)	<40	(>76)	380	(-124)
Iron	1,100	5,000	3,400	(32)	2,400	(52)	1,900	(62)	2,300	(54)
Magnesium	1,700	7,000	6,600	(6)	6,600	(6)	4,900	(30)	6,100	(13)
Manganese	30	20	70	(-250)	40	(-100)	20	(0)	90	(-350)
Molybdenum	<10	<200	<200		<200		<200		<400	
Sodium	2,400	54,000	56,000	(-4)	61,000	(-17)	57,000	(-6)	56,000	(-4)
Silicon	5,600	4,800	3,200	(33)	2,700	(44)	2,200	(54)	2,600	(46)
Tin	<20	<300	<300		<300		<300		<600	
Strontium	95	170	160	(6)	160	(6)	120	(29)	2	(99)
Titanium	<1	200	110	(45)	60	(70)	80	(60)	180	(10)
Vanadium	12	2,700	120	(96)	110	(96)	<40	(>99)	540	(80)
Ammonia nitrogen	NA[b]	3,300	3,000	(9)	2,600	(21)	2,200	(33)	5,500	(-67)
Nitrate nitrogen	NA	5,300	7,100	(-34)	6,500	(-23)	6,500	(-23)	8,800	(-66)
Phosphate phosphorus	NA	200	210	(-5)	100	(50)	100	(50)	160	(20)

[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Not analyzed.

TABLE 20. PLANT W BIOASSAY RESULTS

Test species	Parameter	Secondary effluent		Tertiary effluent			
		Phase I	Phase II	Type 1	Type 3	Type 6	Type 7
Freshwater algae - *S. capricornutum*	EC_{50} - 7 day, % effluent	NM[a]	1.8 (0.6-5.4)[b]	6.0 (0.9-4.0)	12 (3.6-41)	47 (23-95)	37 (26-53)
	EC_{50} - 12 day, % effluent	NM	2.4 (0.5-12)	10 (4.2-24)	17 (1.5-100)	33 (16-68)	44 (5.5-100)
	EC_{50} - 14 day, % effluent	-[c]	4.0 (2.1-7.6)	8.9 (2.6-30)	11 (3.4-37)	28 (13-61)	29 (16-54)
Daphnia - *D. magna*	LC_{50} - 24 hr, % effluent	NM	>36<60	57 (33-99)	58 (34-100)	51 (36-72)	55 (32-95)
	LC_{50} - 48 hr, % effluent	6.3[d]	56 (33-97)	48 (34-66)	58 (34-100)	49 (35-70)	54 (31-93)
Blue gill - *L. macrochirus*	LC_{50} - 24 hr, % effluent	NM	~77	>100	>100	>100	NM
	LC_{50} - 48 hr, % effluent	NM	71 (63-81)	77 (67-85)	68 (61-75)	75 (69-80)	NM
	LC_{50} - 72 hr, % effluent	NM	65 (58-71)	72 (65-81)	61 (54-67)	68 (61-75)	NM
	LC_{50} - 96 hr, % effluent	NM	60 (53-66)	64 (57-71)	60 (53-66)	66 (59-73)	NM
Fathead minnow - *P. promelas*	LC_{50} - 24 hr, % effluent	NM	75 (70-80)	76 (68-89)	>77<100	76 (68-86)	>60<100
	LC_{50} - 48 hr, % effluent	NM	63 (52-76)	59 (50-77)	73 (65-84)	68 (61-75)	68 (54-84)
	LC_{50} - 72 hr, % effluent	NM	61 (51-75)	53 (45-62)	69 (61-79)	65 (58-72)	68 (54-84)
	LC_{50} - 96 hr, % effluent	55	61 (51-75)	48 (39-55)	68 (59-79)	61 (54-67)	65 (51-80)
S. typhimurium - strains TA98, TA100, TA1535, TA1537, and TA1538	Response to Ames test for mutagenicity - (-) or (+)	(-)	(-)	(-)	(-)[e]	(-)	(-)
E. coli - strains W3110 and p3478	Response to pol A test for mutagenicity-increase in zone of inhibition, mm	0	0	0	0	0	0
Chinese hamster ovary cells	Response to CHO-K1 test for acute cytotoxicity-EC_{50}	-[f]	>100	>100	>100	>100	>100

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[a] Not measured.

[b] 95% confidence limits.

[c] Growth of *S. capricornutum* supported with 2% and 5% secondary effluent, but inhibited at 10% and 20% levels.

[d] EC_{50} - 48 hr determined using *Daphnia pulex*.

[e] Increase in number of revertants was observed with TA1538, with and without metabolic activation; however, a dose response was not observed.

[f] EC_{50} not determinable; cytotoxicity procedure employing rabbit alveolar macrophage (RAM) used in Phase I.

TABLE 21. PLANT W EFFLUENT DESCRIPTIONS

Parameter	Secondary effluent		Tertiary effluent			
	Phase I	Phase II	Type 1	Type 3	Type 6	Type 7
Physical description	Cloudy orange with a moderate amount of particulate; nonchlorinated	Cloudy brown liquid	Cloudy brown liquid	Cloudy brown liquid	Pale yellow liquid	Cloudy pale yellow liquid
pH	8.0	7.7	7.8	7.8	7.9	7.6
Salinity, g/ℓ	NM[a]	2	2	2	2	2
Specific conductivity, μmhos/cm^2	NM	2,400	2,900	2,800	2,400	2,900

[a] Not measured.

Plant S

Textile plant S generated wastewater from knit fabric finishing operations. Existing, operating treatment facilities included a gravity separation tank, an equalization basin, an aeration basin, a secondary clarifier, and chlorination. Phase I studies (4) indicated that plant S wastewater treated by the existing treatment facilities was toxic, but ranked eighth when compared to the other 21 secondary effluents evaluated in the program. It was recommended, though, that plant S be included as a site for Phase II tertiary pilot plant studies and MRC sampling/analysis.

After tertiary process screening studies were conducted by Engineering, Science, Inc., it was decided that tertiary treatment types 3, 4, and 6 held the best promise for improving secondary effluent quality to meet BATEA guideline limitations and were thus selected for candidate mode studies. Cationic polymer was used as a precoagulant in the type 4 treatment. A flow diagram of the employed candidate mode configuration is shown in Figure 13. Sampling of the candidate mode operation was conducted by MRC, and results of subsequent analyses and bioassay studies are presented in Tables 22 through 26. In evaluating the data, it is apparent that very few organic toxic pollutants existed in the secondary effluent at levels greater than 10 μg/ℓ. Type 3 treatment (filtration without precoagulation) removed methylene chloride and total phenol while adding bis(2-ethylhexyl) phthalate. Type 4 treatment (filtration with precoagulation) removed bis(2-ethylhexyl) phthalate and methylene chloride while adding total phenol, specific phenols, and cresols. These compounds may have been present in the cationic polymer used as a filter aid or may have leached from the filter media used in type 4 treatment. Type 6 treatment (filtration without precoagulation followed by activated carbon) removed total phenol while adding significant amounts of bis(2-ethylhexyl) phthalate, trichlorofluoromethane, and methylene chloride.

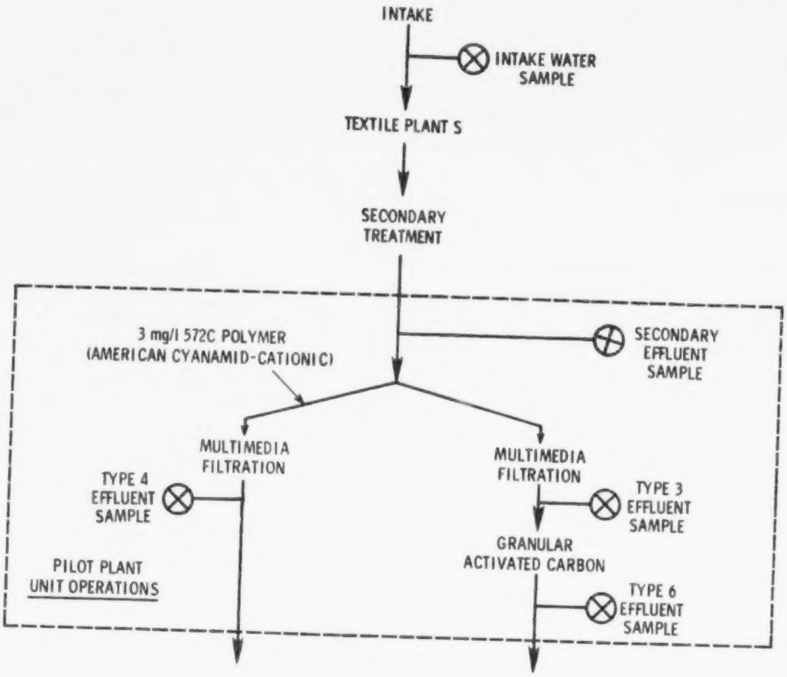
INTAKE
INTAKE WATER SAMPLE
TEXTILE PLANT S
SECONDARY TREATMENT
3 mg/l 572C POLYMER (AMERICAN CYANAMID-CATIONIC)
SECONDARY EFFLUENT SAMPLE
MULTIMEDIA FILTRATION
MULTIMEDIA FILTRATION
TYPE 4 EFFLUENT SAMPLE
TYPE 3 EFFLUENT SAMPLE
GRANULAR ACTIVATED CARBON
PILOT PLANT UNIT OPERATIONS
TYPE 6 EFFLUENT SAMPLE

Figure 13. Candidate wastewater treatment system studied at plant S.

Except for copper and zinc, little change in the concentration of inorganic toxic pollutants occured due to tertiary treatment. Significant amounts of copper (more than 85%) and zinc (24%) were removed by type 6 treatment (activated carbon preceded by filtration), while zinc was added by treatment types 3 and 4. Aluminum, vanadium, and ammonia were removed by all three treatment systems. Iron was added by treatment types 3 and 4, which employ steel filtration columns.

Static acute toxicity testing with freshwater algae was the only bioassay test which indicated the effects of the three treatment types. Toxicity levels remained about the same when type 3 treatment was used; however, the wastewater became more toxic when type 4 treatment was used. All toxicity to freshwater algae was removed by type 6 treatment.

TABLE 22. PLANT S ORGANIC TOXIC POLLUTANTS DETECTED (Concentration, µg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent		Tertiary effluent					
		Phase I	Phase II	Type 3		Type 4		Type 6	
Bis(2-ethylhexyl) phthalate	1.2	41	25	42	(-68)[a]	16	(36)	410	(-1,500)
Acenaphthene	-[b]		2.2	0.6		0.6			
Di-n-butyl phthalate			2.8	6					
Phenol	0.5		0.6	0.4		0.2			
2,4-Dimethylphenol						0.4			
2,4-Dichlorophenol						0.2			
p-Chloro-*m*-cresol						0.3			
Chloroform	120			7					
Toluene	3	21	1.8	0.4		1.4		1.6	
Trichlorofluoromethane								69	(-3,500)
1,2,4-Trichlorobenzene		920							
Naphthalene		260							
Ethylbenzene		110							
Tetrachloroethylene		0.4							
Methylene chloride[c]	55		12	4.6	(62)	7.9	(34)	940	(-7,700)
Phenol (total)	5	29	15	-[d]		21	(-40)	-[d]	
Phenol (total)[e]	-[f]	29	11	9.0	(18)	16	(-45)		(>32)

[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Blanks indicate concentration below detection limit (see Table 6).

[c] Methylene chloride may originate from analysis contamination.

[d] Sample bottle broken in shipment.

[e] Sample taken day after all other samples were taken.

[f] Sample not taken.

TABLE 23. PLANT S INORGANIC TOXIC POLLUTANTS DETECTED (Concentration, µg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent		Tertiary effluent					
		Phase I	Phase II	Type 3		Type 4		Type 6	
Antimony	20	70	610	620	(-2)[a]	600	(2)	590	(3)
Arsenic	<10	<10	<10	<10	(0)	11[b]	(-10)	11[b]	(>10)
Beryllium	<5	<5	<5	<5		<5		<5	
Cadmium	3[b]	2	5[b]	5[b]		6[b]		6[b]	
Chromium	<4	<4	<4	<4		<4		<4	
Copper	10[b]	60	26	27	(-4)	24	(8)	<4	(>85)
Cyanide	<10	<10	<10	<10		<10		<10	
Lead	34[b]	<22	75[b]	81[b]	(-8)	85[b]	(-13)	79[b]	(-5)
Mercury	0.5	<0.3	1.7	0.4		0.7		0.4	
Nickel	61[b]	<36	83[b]	81[b]	(2)	98[b]	(-18)	96[b]	(-16)
Selenium	<10	<10	<10	<10		<10		<10	
Silver	<5	<5	<5	<5		<5		<5	
Thallium	<50	<50	<50	<50		<50		<50	
Zinc	42	84	41	75	(-83)	55	(-34)	31	(24)

[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Semiquantitative region; value not within 95% confidence limits.

TABLE 24. PLANT S OTHER POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent	Tertiary effluent					
			Type 3		Type 4		Type 6	
Aluminum	110	690	450	(35)	330	(52)	310	(55)
Barium	12	8.3	6.2		5.9		5.7	
Boron	<1	1,100	1,100	(0)	1,000	(9)	1,100	(0)
Calcium	5,500	5,900	6,200	(-5)[a]	5,800	(2)	5,200	(12)
Cobalt	8^b	10^b	7^b		11^b	(-10)	6^b	
Iron	240	100	150	(-50)	190	(-90)	58	(42)
Magnesium	860	1,600	1,500	(6)	1,500	(6)	1,400	(13)
Manganese	7.7	11	12	(-9)	11	(0)	8	(27)
Molybdenum	<10	13	14	(-8)	13	(0)	17	(-31)
Sodium	10,000	180,000	190,000	(-6)	180,000	(0)	190,000	(-6)
Silicon	3,600	11,000	11,000	(0)	11,000	(0)	11,000	(0)
Tin	<15	<15	<15		<15		<15	
Strontium	21	22	22	(0)	22	(0)	22	(0)
Titanium	2^b	4^b	4^b		4^b		4^b	
Vanadium	11	57	23	(60)	23	(60)	22	(61)
Ammonia nitrogen	NA[c]	6,600	60	(99)	5,600	(15)	1,200	(82)
Nitrate nitrogen	NA	250	120	(52)	210	(16)	670	(-170)
Phosphate phosphorus	NA	1,700	2,100	(-24)	4,500	(-170)	2,000	(-18)

[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Semiquantitative region; value not within 95% confidence limits.

[c] Not analyzed.

TABLE 25. PLANT S BIOASSAY RESULTS

Test species	Parameter	Secondary effluent		Tertiary effluent		
		Phase I	Phase II	Type 3	Type 4	Type 6
Freshwater algae - *S. capricornutum*	EC_{50} - 7 day, % effluent	NM[a]	79 (5-100)[b]	>100	73 (26-100)	>100
	EC_{50} - 12 day, % effluent	NM	79 (6-100)	40 (6-100)	62 (16-100)	56 (3-100)
	EC_{50} - 14 day, % effluent	-[c]	72 (7-100)	>56<100	49 (25-95)	>100
Daphnia - *D. magna*	LC_{50} - 24 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 48 hr, % effluent	-[d]	>100	>100	>100	>100
Fathead minnow - *P. promelas*	LC_{50} - 24 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 48 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 72 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 96 hr, % effluent	>100	>100	>100	>100	>100
S. typhimurium - strains TA98, TA100, TA1535, TA1537, and TA1538	Response to Ames test for mutagenicity - (-) or (+)	(-)	(-)	(-)	(-)	(-)
E. coli - strains W3110 and p3478	Response to pol A test for mutagenicity-increase in zone of inhibition, mm	0	0	0	0	0
Chinese hamster ovary cells	Response to CHO-K1 test for acute cytotoxicity-EC_{50}	NM	>100	>100	>100	>100

[a] Not measured.

[b] 95% confidence interval.

[c] 20% secondary effluent was highly stimulatory to the growth of *S. capricornutum*.

[d] LC_{50} not calculated since a heavy solids concentration abscured the analysis; sample did not appear to be acutely toxic; *Daphnia pulex* used in determination.

TABLE 26. PLANT S EFFLUENT DESCRIPTIONS

Parameter	Secondary effluent		Tertiary effluent		
	Phase I	Phase II	Type 3	Type 4	Type 6
Physical description	Clear, light champagne with small amount of particulate; unchlorinated	Orange liquid containing a precipitate	Orange liquid containing a precipitate	Light orange liquid containing a precipitate	Clear liquid
pH	7.7	7.2	7.3	7.0	7.4
Salinity, g/ℓ	NM[a]	1	0	1	1
Specific conductivity, μmhos/cm³	NM	1,100	870	1,100	1,100

[a] Not measured.

Plant P

Textile plant P generated wastewater from knit fabric finishing operations. Existing treatment facilities included screens, aeration, secondary clarification, and chlorination. Phase I screening studies (4) indicated that plant P wastewater treated by the existing facilities ranked ninth in terms of toxicity when compared to the other 21 secondary effluents evaluated in the program. Therefore, plant P was included as a site for Phase II tertiary pilot plant studies and MRC sampling/analysis.

After tertiary process screening studies were conducted by Engineering Science, Inc., it was decided that tertiary treatment types 2, 3, 5, and 6 held the best promise of improving secondary effluent quality to meet suggested BATEA guidelines; they were thus selected for candidate mode studies. Cationic polymer was used as a coagulant in treatment types 2 and 5. A flow diagram of the candidate mode configuration used is shown in Figure 14. Sampling of the candidate mode operation was conducted by MRC, and results of subsequent analysis and bioassay work are presented in Tables 27 through 31. Organic toxic pollutant analysis provided few data on relative removal efficiencies because few such pollutants were found in the secondary effluent. Only total phenol existed at a level greater than 10 μg/ℓ. This was removed by type 6 treatment (activated carbon preceded by filtration).

Significant (more than 38%) removals of antimony, chromium, copper, and zinc were attained by all four treatment systems. Arsenic was apparently added by type 6 treatment (activated carbon preceded by filtration). Aluminum, iron, manganese, molybdenum, titanium, and vanadium were also removed by all four treatment systems.

Again, only freshwater algae toxicity studies indicated the detrimental effects of the wastewaters to bioassay test species. Only type 6 treatment improved the effluent quality in terms of freshwater algae toxicity.

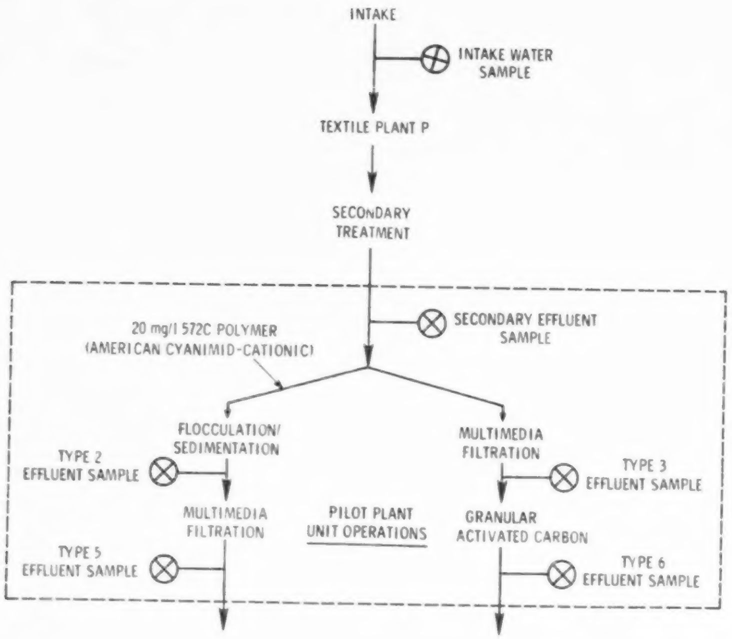
INTAKE
INTAKE WATER SAMPLE
TEXTILE PLANT P
SECONDARY TREATMENT
SECONDARY EFFLUENT SAMPLE
20 mg/l 572C POLYMER (AMERICAN CYANIMID-CATIONIC)
FLOCCULATION/ SEDIMENTATION
MULTIMEDIA FILTRATION
TYPE 2 EFFLUENT SAMPLE
TYPE 3 EFFLUENT SAMPLE
MULTIMEDIA FILTRATION
PILOT PLANT UNIT OPERATIONS
GRANULAR ACTIVATED CARBON
TYPE 5 EFFLUENT SAMPLE
TYPE 6 EFFLUENT SAMPLE

Figure 14. Candidate wastewater treatment system studied at plant P.

TABLE 27. PLANT P ORGANIC TOXIC POLLUTANTS DETECTED (Concentration, μg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent Phase I	Secondary effluent Phase II	Tertiary effluent Type 2	Tertiary effluent Type 3	Tertiary effluent Type 5	Tertiary effluent Type 6
Bis(2-ethylhexyl) phthalate	2.0	72	10	10	3.9	3.3	3.9
Di-n-butyl phthalate	0.9	–[a]	2.1	2.8	1.6	2.5	
Diethyl phthalate			1.3		0.8	1.0	1.4
Anthracene			0.8	0.9	0.5	0.5	0.1
Phenol			0.7	0.5	1.8	2.6	
Chloroform	4.1	6.9					
Trichloroethylene	1.4			0.8			
Toluene	1.5	22	0.4	0.4	2.7	2.6	3.6
Benzene				0.4	1.0	0.5	
N-nitroso-di-n-propylamine		19					
Ethylbenzene		280		0.1			
Methylene chloride[b]	20		0.4	2.5	4.1	4.7	7.3
Phenol (total)	11	32	72	82 (-14)[c]	68 (6)	130 (-81)	18 (75)

[a] Blanks indicate concentration below detection limit (see Table 6).

[b] Methylene chloride may originate from analysis contamination.

[c] Minus percent removals indicate an increase in the concentration of the specified pollutant.

TABLE 28. PLANT P INORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

		Secondary effluent		Tertiary effluent							
Pollutant	Intake	Phase I	Phase II	Type 2		Type 3		Type 5		Type 6	
Antimony	28^a	<10	77^a	43^a	(44)	48^a	(38)	34^a	(56)	36^a	(53)
Arsenic	<2	<2	<2	<2		<2		<2		12	$(-500)^b$
Beryllium	<0.2	<0.1	<0.2	<0.2		<0.2		<0.2		<0.2	
Cadmium	<2	<2	<2	<2		<2		<2		<2	
Chromium	5.7^a	<4	98	<4	(>96)	<4	(>96)	<4	(>96)	<4	(>96)
Copper	98	<4	36	<4	(>89)	<4	(>89)	<4	(>89)	<4	(>89)
Cyanide	<4	140	<4	<4		<4		<4		<4	
Lead	58^a	<22	25^a	<22	(>12)	<22	(>12)	<22	(>12)	<22	(>12)
Mercury	<0.3	<0.5	0.4	<0.3		0.3		0.4		0.4	
Nickel	56^a	40	66^a	43^a	(35)	58^a	(12)	36^a	(83)	50^a	(24)
Selenium	<8	<5	<8	<8		<8		<8		<8	
Silver	<5	8	<5	<5		5		<5		<5	
Thallium	<50	<50	<50	<50		<50		<50		<50	
Zinc	150	140	5,200	160	(97)	150	(97)	160	(97)	<1	(100)

[a] Semiquantitative region; value not within 95% confidence limits.

[b] Minus percent removals indicate an increase in the concentration of the specified pollutant.

TABLE 29. PLANT P OTHER POLLUTANTS DETECTED
(Concentration, µg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent		Tertiary effluent							
		Phase I	Phase II	Type 2		Type 3		Type 5		Type 6	
Aluminum	670	140	300	20[a]	(93)	30[a]	(90)	50[a]	(83)	40[a]	(87)
Barium	11	<0.2	<0.2	<0.2		<0.2		<0.2		<0.2	
Boron	150	520	950	880	(7)	860	(9)	880	(7)	660	(31)
Calcium	3,600	9,500	8,300	7,500	(10)	7,500	(10)	7,300	(10)	5,000	(40)
Cobalt	<6	0.5	<6	<6		<6		<6		<6	
Iron	1,400	100	1,000	300	(70)	300	(70)	600	(40)	20	(98)
Magnesium	1,600	1,800	1,800	1,800	(0)	1,800	(0)	1,800	(0)	1,500	(17)
Manganese	73	20	80	49	(39)	50	(38)	43	(46)	<0.5	(>99)
Molybdenum	10[a]	<0.6	20[a]	<10	(>50)	<10	(>50)	<10	(>50)	<10	(>50)
Sodium	6,200	>100,000	130,000	120,000	(8)	120,000	(8)	120,000	(8)	110,000	(15)
Silicon	6,400	4,800	2,400	2,300	(4)	2,300	(4)	2,100	(13)	2,600	(-8)
Tin	16[a]	<10	<15	22[a]	(-40)[b]	<15		18[a]	(-20)	<15	
Strontium	25	NA[c]	35	33	(6)	33	(6)	35	(0)	35	(0)
Titanium	36	<1	36	<1	(>97)	<1	(>97)	<1	(>97)	<1	(>97)
Vanadium	21	20	18	14	(22)	15	(17)	15	(17)	14	(22)
Ammonia nitrogen	NA	200	790	700	(11)	360	(54)	580	(27)	830	(-5)
Nitrate nitrogen	NA	80	300	280	(7)	130	(57)	250	(17)	300	(0)
Phosphate phosphorus	NA	20[d]	2,800	2,900	(-4)	2,700	(4)	2,500	(11)	2,400	(14)

[a] Semiquantitative region; value not within 95% confidence limits.

[b] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[c] Not analyzed.

[d] *o*-Phosphate only.

TABLE 30. PLANT P BIOASSAY RESULTS

Test species	Parameter	Secondary effluent		Tertiary effluent			
		Phase I	Phase II	Type 2	Type 3	Type 5	Type 6
Freshwater algae - *S. capricornutum*	EC_{50} - 7 day, % effluent	NM[a]	54 (30-100)[b]	41 (14-100)	42 (29-62)	64 (25-100)	83 (18-100)
	EC_{50} - 12 day, % effluent	NM	54 (18-100)	24 (13-42)	42 (14-100)	>32<100	>56<100
	EC_{50} - 14 day, % effluent	-[c]	53 (20-100)	26 (15-43)	33 (10-100)	41 (28-59)	>56<100
Daphnia - *D. magna*	LC_{50} - 24 hr, % effluent	NM	>100	>100	>100	>100	>100
	LC_{50} - 48 hr, % effluent	>100[d]	>100	>100	>100	>100	>100
Fatheat minnow - *P. promelas*	LC_{50} - 24 hr, % effluent	NM	-[e]	-[e]	-[e]	-[e]	-[e]
	LC_{50} - 48 hr, % effluent	NM	-[e]	-[e]	-[e]	-[e]	-[e]
	LC_{50} - 72 hr, % effluent	NM	-[e]	-[e]	-[e]	-[e]	-[e]
	LC_{50} - 96 hr, % effluent	>100	-[e]	-[e]	-[e]	-[e]	-[e]
S. typhimurium - strains TA98, TA100, TA1535, TA1537 and TA1538	Response to Ames test for mutagenicity (-) or (+)	(-)	(-)	(-)	(-)	(-)	(-)
Chinese hamster ovary cells	Response to CHO-K1 test	>100	>100	>100	>100	>100	>100

[a] Not measured.

[b] 95% confidence interval.

[c] 20% secondary effluent stimulated the growth of *S. capricornutum*.

[d] EC_{50} - 48 hr determined with *Dphnia pulex*.

[e] LC_{50}'s not calculated because of data scatter; however, none of the samples appear to be toxic since there was no more than a 20% kill in any of the tests with 100% effluent.

TABLE 31. PLANT P EFFLUENT DESCRIPTIONS

	Secondary effluent		Tertiary effluent			
Parameter	Phase I	Phase II	Type 2	Type 3	Type 5	Type 6
Physical description	NM[a]	Brown turbid liquid containing suspended particulate matter	Brown turbid liquid, suspended particulate matter present	Brown turbid liquid, suspended particulate matter present	Brown turbid liquid suspended particulate matter present	Slightly cloudy pale yellow liquid
pH	NM	6.6	6.9	6.8	6.8	6.9
Salinity, g/ℓ	NM	0	0	0	0	0
Specific conductivity, μmhos/cm²	NM	600	520	500	490	480

[a] Not measured.

Plant N

Textile plant N generated wastewater from wool finishing operations. Existing treatment facilities included screens, aeration, and secondary clarification. Phase I screening studies (4), indicated that plant N wastewater treated by the existing facilities was 1 of the more toxic effluents of the 22 secondary effluents evaluated in the program. Therefore, plant N was included as a site for Phase II tertiary pilot plant studies and MRC sampling/analysis.

After tertiary process screening studies were conducted by Engineering Science, Inc., it was decided that tertiary treatment types 3, 5, and 6 held the best promise for improving secondary effluent quality to suggested BATEA guidelines, and they were selected for candidate mode studies. Alum was used as a coagulant and NaOH used for adjusting pH in treatment type 5. Figure 15 is a flow diagram of the candidate mode configuration employed.

Sampling of the candidate mode operation was conducted by MRC, and results of subsequent chemical analyses and bioassay studies are presented in Tables 32 through 36. In evaluating organic toxic pollutant data, it can be seen, as has been seen in data of former plants, that only very few organic toxic pollutants occurred in the secondary effluent. Those that did occur include bis(2-ethylhexyl) phthalate, methylene chloride, and total phenol. Type 3 treatment (filtration) removed bis(2-ethylhexyl) phthalate and total phenol, while treatment 5 and 6 reduced levels of bis(2-ethylhexyl) phthalate, methylene chloride, and total phenol.

Antimony, chromium, cobalt, iron, zinc, and sulfide were removed by all three treatment types, while copper was added. As expected, aluminum was added by type 5 treatment, where alum was used as a coagulant, and removed by treatment types 3 and 6. Phosphorus was removed by treatment types 5 and 6.

Type 5 treatment (filtration preceded by flocculation/sedimentation) was effective in reducing toxicity to *Daphnia*. The tertiary technologies employed had little effect upon effluent toxicity to other bioassay species.

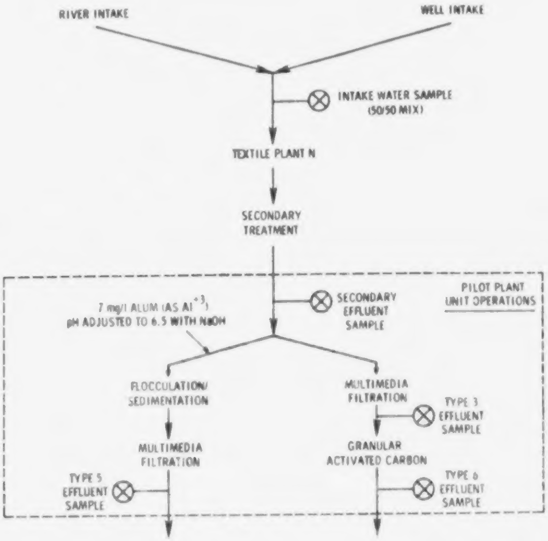
RIVER INTAKE
WELL INTAKE
INTAKE WATER SAMPLE (50/50 MIX)
TEXTILE PLANT N
SECONDARY TREATMENT
PILOT PLANT UNIT OPERATIONS
7 mg/l ALUM (AS Al+3)
pH ADJUSTED TO 6.5 WITH NaOH
SECONDARY EFFLUENT SAMPLE
FLOCCULATION/ SEDIMENTATION
MULTIMEDIA FILTRATION
TYPE 3 EFFLUENT SAMPLE
MULTIMEDIA FILTRATION
GRANULAR ACTIVATED CARBON
TYPE 5 EFFLUENT SAMPLE
TYPE 6 EFFLUENT SAMPLE

Figure 15. Candidate wastewater treatment system studied at plant N.

TABLE 32. PLANT N ORGANIC TOXIC POLLUTANTS DETECTED (Concentration, µg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent		Tertiary effluent					
		Phase I	Phase II	Type 3		Type 5		Type 6	
Bis(2-ethylhexyl) phthalate	53	16.7	230	29	(87)	31	(87)	78	(66)
Anthracene	0.2	–[a]	0.4			0.4		0.4	
Diethyl phthalate	1.0	9.4	0.8	0.4		0.3		1.2	
Di-n-butyl phthalate	1.2		0.6	1.1		0.6		1.8	
Methylene chloride[b]	47		46	47	(-2)[c]	28	(39)	27	(41)
Toluene	0.9	17	0.4	0.6		0.4			
1,2-Dichlorobenzene		6.0	0.9			0.5			
Dimethyl phthalate			1.4						
Fluoranthene			0.07	0.08		0.05			
Pyrene			0.1	0.1		0.09			
Fluorene						0.05			
2,4-Dichlorophenol						0.5			
Phenanthrene				0.5					
1,2-Dichloropropane				1.0					
1,4-Dichlorobenzene		1.5							
Tetrachloroethylene			0.7						
Ethylbenzene		75	0.9						
Phenol		11							
2,4-Dimethylphenol		8							
Phenol (total)	14	68	31	17	(45)	25	(19)	11	(65)

[a] Blanks indicate concentration below detection limit (see Table 6).

[b] Methylene chloride may originate from analysis contamination.

[c] Minus percent removals indicate an increase in the concentration of a specified pollutant.

TABLE 33. PLANT N INORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

		Secondary effluent		Tertiary effluent					
Pollutant	Intake	Phase I	Phase II	Type 3		Type 5		Type 6	
Antimony	14[a]	<10	18[a]	<10	(>44)	<10	(>44)	<10	(>44)
Arsenic	5	<5	3	3		<1		3	
Beryllium	0.6	NA[b]	<0.04	<0.04		0.1[a]		<0.04	
Cadmium	<2	<0.5	<2	<2		<2		<2	
Chromium	<4	1,800	170	95	(44)	34	(80)	5.2[a]	(97)
Copper	160	8	14[a]	130	(-830)[c]	86	(-510)	24	(-71)
Cyanide	<5	<4	<5	<5		<5		<5	
Lead	<22	<1	<22	<22		<22		<22	
Mercury	<0.1	<0.5	<0.1	<0.1		<0.1		<0.1	
Nickel	<36	30	<36	<36		<36		<36	
Selenium	<1	<5	<1	<1		<1		<1	
Silver	10[a]	<5	5.5[a]	<5		<5		<5	
Thallium	<50	<5	<50	<50		<50		<50	
Zinc	22	38,000	1,300	590	(55)	440	(66)	430	(67)

[a] Semiquantitative region; value not within 95% confidence limits.

[b] Not analyzed.

[c] Minus percent removals indicate an increase in the concentration of the specified pollutant.

TABLE 34. PLANT N OTHER POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

Pollutant	Intake	Secondary effluent	Tertiary effluent					
			Type 3		Type 5		Type 6	
Aluminum	120	120	36[a]	(67)	390	(-260)[b]	21[a]	(81)
Barium	6.7	6.0	3.3		2.7		4.8	
Boron	0.6	9.1	6.7		6.4		9.0	
Calcium	5,400	7,100	6,200	(13)	6,200	(13)	5,000	(30)
Cobalt	6.2[a]	12[a]	8.7[a]	(28)	8.6[a]	(28)	<6	(>50)
Iron	620	720	290	(60)	55	(92)	110	(85)
Magnesium	1,000	1,100	990	(10)	1,000	(9)	860	(22)
Manganese	530	210	190	(10)	200	(5)	160	(24)
Molybdenum	<10	<10	<10		<10		<10	
Sodium	12,000	180,000	180,000	(0)	200,000	(-11)[b]	180,000	(0)
Phosphorus	<70	2,500	2,300	(8)	1,200	(52)	1,000	(60)
Silicon	4,000	3,800	3,700	(3)	3,400	(11)	3,800	(0)
Tin	<15	<15	<15		<15		<15	
Strontium	51	41	40	(2)	39	(5)	40	(2)
Titanium	2.1[a]	1.9[a]	1.2[a]		<1		<1	
Vanadium	6.9[a]	8.5[a]	7.7[a]		8.5[a]		6.5[a]	
Sulfide	14	12	<5	(>58)	<5	(>58)	<5	(>58)
Ammonia nitrogen	NA[c]	1,700	1,200	(29)	1,600	(6)	1,300	(24)
Nitrate nitrogen	NA	5,200	4,600	(12)	5,200	(0)	3,100	(40)
COD	11,000	128,000	210,000	(-64)	172,000	(-34)	44,000	(66)
TSS	6,000	75,000	<1,000	(>99)	8,000	(89)	12,000	(84)
Color[d] at pH 7.6	47	39	38	(3)	52	(33)	51	(-31)
Color[d] at sample pH	48	36	43	(-19)	48	(-33)	51	(-42)
pH[e]	6.46	7.97	6.95		7.45		746	(6.4)

[a] Semiquantitative region; value not within 95% confidence limits.

[b] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[c] Not analyzed.

[d] ADMI units.

[e] pH units (- log [H+]).

TABLE 35. PLANT N BIOASSAY RESULTS

Test species	Parameter	Secondary effluent		Tertiary effluent		
		Phase I	Phase II	Type 3	Type 5	Type 6
Freshwater algae - *S. capricornutum*	EC_{50} - 7 day, % effluent	NM[a]	20 (11-36)[b]	23 (13-41)	44 (26-75)	23 (10-51)
	EC_{50} - 12 day, % effluent	NM	28 (20-41)	38 (27-53)	35 (19-66)	16 (10-27)
	EC_{50} - 14 day, % effluent	-[c]	23 (18-31)	30 (16-56)	29 (15-57)	23 (14-38)
Daphnia - *D. magna*	LC_{50} - 24 hr, % effluent	NM	>100 (86->100)	>100	>100	>100
	LC_{50} - 48 hr, % effluent	-[d]	77 (60-100)	78 (67-92)	>100	77 (60-100)
Fathead minnow - *P. promelas*	LC_{50} - 24 hr, % effluent	NM	>100	>100	>100	>100
	LC_{50} - 48 hr, % effluent	NM	91 (73->100)	>100	>100	>100
	LC_{50} - 72 hr, % effluent	NM	81 (68-99)	>100	>100	>100
	LC_{50} - 96 hr, % effluent	49	81 (68-99)	>100	>100	>100
S. typhimurium - strains TA98, TA100, TA1535, TA1537, and TA1538	Response to Ames test for mutagenicity - (-) or (+)	(-)	(-)	(-)	(-)	(-)
E. coli - strains W3110 and p3478	Response to pol A test for mutagenicity-increase in zone of inhibition, mm	0	0	0	0	0
Chinese hamster ovary cells	Response to CHO-K1 test for acute cytotoxicity-EC_{50}	-[e]	>100	>100	>100	>100

[a] Not measured.

[b] 95% confidence interval.

[c] All concentrations of secondary effluent (2%, 5%, 10%, and 10%) failed to support the growth of *S. capricornutum*.

[d] 100% kill in all dilutions (4.7% - 100%), EC_{50} determined with *Daphnia pulex*.

[e] EC_{50} not determinable; cytotoxicity procedure employing rabbit alveolar macrophage (RAM) used in Phase I.

TABLE 36. PLANT N EFFLUENT DESCRIPTION

Parameter	Secondary effluent		Tertiary effluent		
	Phase I	Phase II	Type 3	Type 5	Type 6
Physical description	Clear, light grey liquid with moderate amount of particulate matter; non-chlorinated	Turbid, brown liquid with suspended particles	Turbid, brown liquid with suspended particles	Cloudy, light brown liquid with suspended particles	Slightly turbid liquid
pH	3.7	6.6	6.7	6.9	6.5
Salinity, g/ℓ	NM	0	0	0	0
Specific conductivity, μmhos/cm²	NM	600	650	650	850

Plant V

Textile plant V generated wastewater from woven fabric finishing operations. Existing treatment facilities included bar screens, aeration, secondary clarification, and chlorination. Phase I screening studies (4) indicated that plant V wastewater treated by the existing treatment facilities was 1 of the more toxic effluents of the 22 secondary effluents evaluated in the program. Therefore, plant V was included as a site for Phase II tertiary pilot plant studies and MRC sampling/analysis.

After tertiary process screening studies were conducted by Engineering Science, Inc., it was decided that tertiary treatment types 3, 4, 6, and 7 held the best promise of meeting suggested BATEA guidelines and were thus selected for candidate mode studies. Ferric chloride was used as a precoagulant in type 4 treatment. A flow diagram of the employed candidate mode configuration is shown in Figure 16. Sampling of the candidate mode operation was conducted by MRC, and results of subsequent analysis and bioassay work are presented in Tables 37 through 41. As shown in Table 37, bis(2-ethylhexyl) phthalate was added to the secondary effluent by all four treatment systems, while methylene chloride and total phenol were removed. Treatment type 3 (filtration without precoagulation) also added some di-n-butyl phthalate.

Treatment type 3 removed cyanide and small amounts of copper and zinc while adding only lead, while type 4 treatment added seven inorganic toxic pollutants to the secondary effluent. Type 6 treatment removed additional copper, cyanide, and zinc while adding antimony, lead, nickel, and silver. Type 7 treatment removed only cyanide while adding four other inorganic toxic pollutants.

A significant amount of iron (6,000 μg/ℓ) was added to the wastewater by type 4 treatment, since ferric chloride was used as a precoagulant. Aluminum, cobalt, and manganese were also added by type 4 treatment, but they were not added by the other three treatment systems. As expected, type 4 treatment removed

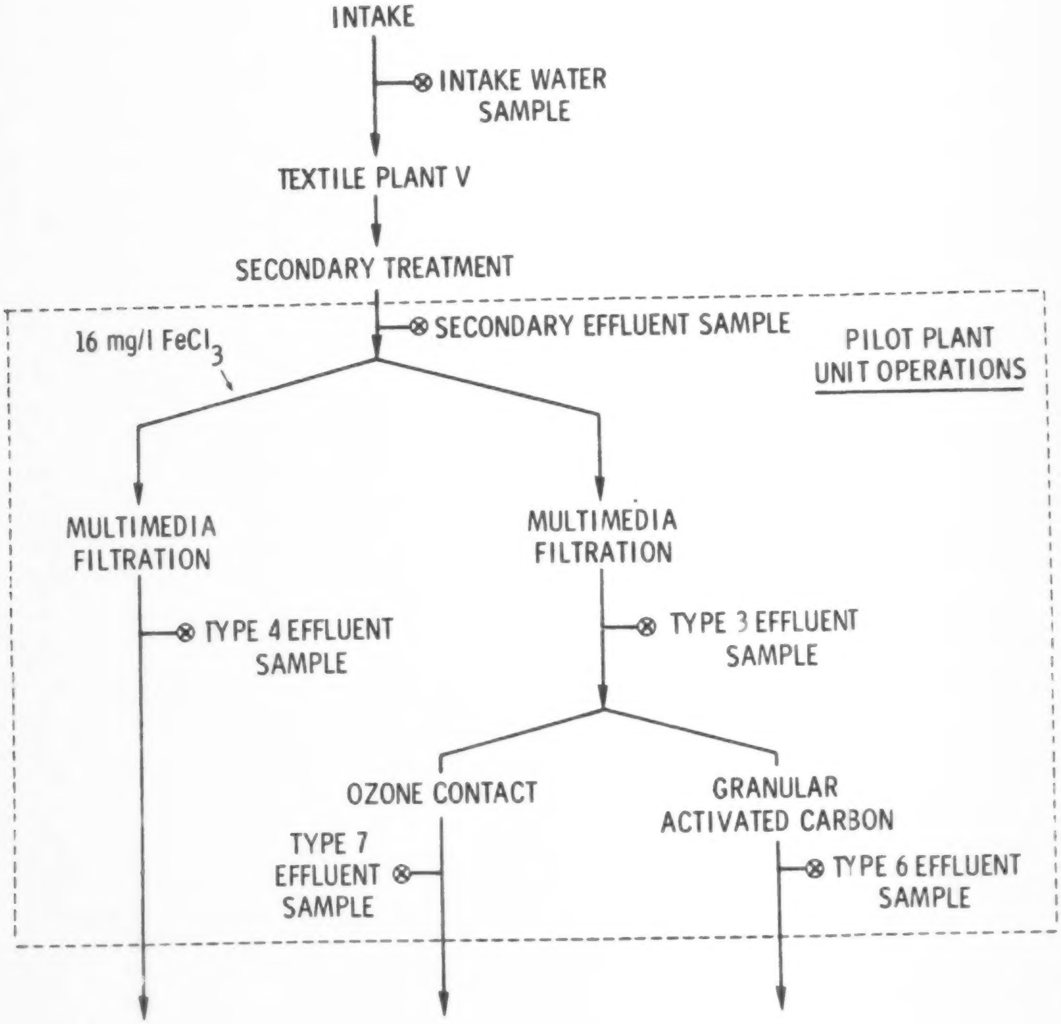
INTAKE
INTAKE WATER SAMPLE
TEXTILE PLANT V
SECONDARY TREATMENT
SECONDARY EFFLUENT SAMPLE
PILOT PLANT UNIT OPERATIONS
16 mg/l FeCl3
MULTIMEDIA FILTRATION
MULTIMEDIA FILTRATION
TYPE 4 EFFLUENT SAMPLE
TYPE 3 EFFLUENT SAMPLE
OZONE CONTACT
GRANULAR ACTIVATED CARBON
TYPE 7 EFFLUENT SAMPLE
TYPE 6 EFFLUENT SAMPLE

Figure 16. Candidate wastewater treatment system studied at plant V.

phosphorus, since iron coagulation used in type 4 treatment is a classical phosphorus removal method.

By reviewing Table 40, it is apparent that type 4 treatment (filtration with precoagulation) was detrimental to freshwater algae, and *Daphnia*. All other treatment series (3, 6, and 7) maintained the original toxic level of the secondary effluent.

TABLE 37. PLANT V ORGANIC TOXIC POLLUTANTS DETECTED

Pollutant	Intake	Secondary effluent		Tertiary effluent							
		Phase I	Phase II	Type 3		Type 4		Type 6		Type 7	
Bis(2-ethylhexyl) phthalate	17	9.5	9.5	16	(-68)[a]	46	(-380)	17	(-79)	90	(-850)
Di-n-butyl phthalate	2.0	-[b]	5.7	12	(-110)	5.4				2.7	
Anthracene			0.2	0.3		0.1					
Butyl benzyl phthalate				0.9							
Methylene chloride[c]	18		24	13	(46)	14	(42)	17	(29)	15	(38)
Toluene	1.3	1,400	1.1	1.3		1.1		1.0		0.9	
Trichloroethylene	1.5		0.7	0.4		2.1		0.6		0.9	
1,1-Dichloroethane	0.4										
Benzene	0.05			0.05							
Ethylbenzene	0.07							0.1		0.1	
Chloroform						0.2					
Trans-1,2-dichloroethylene								1.1		2.1	
Phenol (total)	1,000	16	29	13	(55)	22	(24)	8	(72)	21	(28)

[a] Minus percent removals indicate any increase in the concentration of the specified pollutant.

[b] Blanks indicate concentration below detection limit (see Table 6).

[c] Methylene chloride may originate from analysis contamination.

TABLE 38. PLANT V INORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

		Secondary effluent		Tertiary effluent							
Pollutant	Intake	Phase I	Phase II	Type 3		Type 4		Type 6		Type 7	
Antimony	<10	4	<10	<10		24[a]	(-140)[b]	24[a]	(-140)	25[a]	(-150)
Arsenic	<1	<5	4	4		<1		5		4	
Beryllium	<0.04	<0.1	<0.04	<0.04		<0.04		<0.04		<0.04	
Cadmium	<2	<0.5	<2	<2		<2		<2		<2	
Chromium	<4	3	4.3[a]	<4		6.7[a]		<4		6.3[a]	
Copper	<4	170	85	75	(12)	100	(-18)	16[a]	(81)	89	(-5)
Cyanide	<2	18	23	3	(87)	27	(-17)	<2	(>91)	<2	(>91)
Lead	<22	<1	<22	31[a]	(-41)	37[a]	(-68)	26[a]	(-18)	<22	
Mercury	<1.1	<0.5	<1.1	<1.1		<1.1		<1.1		<1.1	
Nickel	<36	<10	<36	<36		73[a]	(-103)	67[a]	(-86)	66[a]	(-83)
Selenium	<1	<5	<1	<1		<1		2		<1	
Silver	<5	<5	<5	<5		12[a]	(-140)	15[a]	(-200)	16[a]	(-220)
Thallium	<50	<5	<50	<50		<50		<50		<50	
Zinc	98	340	240	190	(21)	330	(-38)	69	(71)	240	(0)

[a] Semiquantitative region; value not within 95% confidence limits.

[b] Minus percent removals indicate an increase in the concentration of the specified pollutant.

TABLE 39. PLANT V OTHER POLLUTANTS DETECTED
(Concentration, µg/ℓ; percent removal in parentheses)

			Tertiary effluent							
Pollutant	Intake	Secondary effluent	Type 3		Type 4		Type 6		Type 7	
Aluminum	380	128	70	(45)	520	(-306)[a]	100	(22)	130	(-2)
Barium	23	14	13	(7)	18	(-29)	21	(-33)	13	(7)
Boron	3.1[b]	730	740	(1)	730	(0)	730	(0)	740	(-1)
Calcium	3,700	5,100	4,500	(12)	5,000	(2)	4,400	(14)	4,800	(6)
Cobalt	6.1[b]	<6	<6		21[b]	(-250)	<6		7.6	
Iron	2,200	210	210	(0)	6,200	(-2,800)	160	(24)	250	(-19)
Magnesium	1,400	2,200	2,200	(0)	2,200	(0)	2,000	(9)	2,200	(0)
Manganese	150	77	80	(-4)	190	(-150)	36	(53)	73	(5)
Molybdenum	<10	<10	<10		<10		<10		<10	
Sodium	3,900	54,000	54,000	(0)	53,000	(2)	51,000	(6)	53,000	(2)
Phosphorus	70	1,200	1,100	(8)	230[a]	(81)	1,100	(8)	1,100	(8)
Silicon	5,900	4,600	4,800	(-4)	4,800	(-4)	4,800	(-4)	4,700	(-2)
Tin	<15	<15	<15		<15		<15		<15	
Strontium	30	31	31	(0)	33	(-6)	27	(13)	31	(0)
Titanium	20	1.4[b]	1.0		2.2[b]		1.9[b]		2.1[b]	
Vanadium	10	13	14	(-8)	21	(-62)	22	(-69)	16	(-23)
Ammonia nitrogen	NA[c]	420	220	(48)	560	(-33)	210	(50)	260	(38)
Nitrate nitrogen	NA	1,300	3,400	(-160)	2,000	(-54)	2,000	(-54)	1,800	(-38)
COD	9,500	93,000	72,000	(23)	36,000	(61)	22,000	(76)	76,000	(18)
TSS	38,000	12,000	4,000	(67)	20,000	(-67)	6,000	(50)	12,000	(0)
Sulfide	<3	<3	<3		<3		<3		<3	
Color[d] at pH 7.6	47	180	180	(0)	43	(76)	30	(83)	130	(28)
Color[d] at sample pH	46	180	182	(-1)	34	(81)	31	(83)	140	(22)
pH[e]	7.3	7.7	7.6		4.0		7.8		7.4	

[a] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[b] Semiquantitative region; value not within 95% confidence limit.

[c] Not analyzed.

[d] ADMI units.

[e] pH units (- log [H$^+$]).

TABLE 40. PLANT V BIOASSAY RESULTS

Test species	Parameter	Secondary effluent		Tertiary effluent			
		Phase I	Phase II	Type 3	Type 4	Type 6	Type 7
Freshwater algae - *S. capricornutum*	EC_{50} - 7 day, % effluent	NM[a]	78 (59-100)[b]	76 (60-96)	19 (14-27)	>100	>100
	EC_{50} - 12 day, % effluent	NM	94 (57-100)	95 (52-100)	25 (13-47)	>100	>100
	EC_{50} - 14 day, % effluent	-[c]	>100	>100	24 (12-48)	>100	>100
Daphnia - *D. magna*	LC_{50} - 24 hr, % effluent	NM	>100	>100	77 (60-100)	>100	>100
	LC_{50} - 48 hr, % effluent	9.4[d]	>60<100	>100	54 (44-66)	>100	>100
Fathead minnow - *P. promelas*	LC_{50} - 24 hr, % effluent	NM	-[e]	-[e]	-[e]	-[e]	-[e]
	LC_{50} - 48 hr, % effluent	NM	-[e]	-[e]	-[e]	-[e]	-[e]
	LC_{50} - 72 hr, % effluent	NM	-[e]	-[e]	-[e]	-[e]	-[e]
	LC_{50} - 96 hr, % effluent	36	-[e]	-[e]	-[e]	-[e]	-[e]
S. typhimurium - strains TA98, TA100, TA1535, TA1537, and TA1538	Response to Ames test for mutagenicity- (-) or (+)	(-)	(-)	(-)	(-)	(-)	(-)
E. coli - strains W3110 and p3478	Response to pol A test for mutagenicity-increase in zone of inhibition, mm	0	0	0	0	0	0
Chinese hamster ovary cells	Response to CHO-K1 test for acute cytotoxicity- EC_{50}, % effluent	-[f]	>100	>100	>100	>100	>100

[a] Not measured.

[b] 95% confidence limits.

[c] 20% secondary effluent was highly stimulatory for the growth of *S. capricornutum*.

[d] EC_{50} determined with *Daphnia pulex*.

[e] LC_{50} not calculated since mortality data did not follow a normal dose - response relationship; secondary effluent (Phase II), Type 3 tertiary effluent, Type 7 tertiary effluent, and Type 8 tertiary effluent do not appear to be acutely toxic to the fathead minnow.

[f] EC_{50} not determinable; cytotoxicity procedure employing rabbit alveolar macrophage (RAM) used in Phase I.

TABLE 41. PLANT V EFFLUENT DESCRIPTIONS

Parameter	Secondary effluent		Tertiary effluent			
	Phase I	Phase II	Type 3	Type 4	Type 6	Type 7
Physical description	NM[a]	Turbid dark brown liquid	Turbid dark brown liquid	Turbid, tan liquid with suspended particles	Slightly turbid liquid	Turbid brown liquid, particles present
pH	NM	6.5	6.3	3.5	6.6	6.5
Salinity, g/ℓ	NM	0	0	0	0	0
Specific conductivity, µmhos/cm²	NM	210	220	300	210	220

[a] Not measured.

Plant T

Textile plant T generated wastewater from woven fabric finishing operations. Existing treatment facilities included aerated equalization, and an aeration basin. Phase I screening studies (4) indicated that plant T wastewater treated by the existing treatment facilities was 1 of the more toxic effluents of the 22 secondary effluents evaluated in the program. Therefore, plant T was included as a site for Phase II tertiary pilot plant studies and MRC sampling/analysis.

After tertiary process screening studies were conducted by Engineering Science, Inc., it was decided that tertiary treatment types 2, 3, 5, and 6 held the best promise for meeting suggested BATEA guidelines; they were thus selected for candidate mode studies. Alum was used as a coagulant and NaOH was used for adjusting pH in treatment types 2 and 5. A flow diagram of the employed candidate mode configuration is shown in Figure 17. Sampling of the candidate mode operation, with the exception of type 2 effluent, was conducted by MRC, and results of subsequent analyses and bioassay studies are presented in Tables 42 through 46. In evaluating the organic toxic pollutant data shown in Table 42 it can be seen that only three pollutants exist in the secondary effluent in concentrations greater than 10 µg/ℓ. Significant amounts of bis(2-ethylhexyl) phthalate and total phenol were removed by type 5 treatment (flocculation/sedimentation followed by filtration). Total phenol was added by treatment types 3 (filtration) and 6 (filtration followed by granular activated carbon), but these treatment systems also removed some bis(2-ethylhexyl) phthalate.

In evaluating the inorganic toxic pollutant data shown in Table 43, it is apparent that treatment type 5 (flocculation/sedimentation followed by filtration) provided the best removal of inorganic toxic species. Type 5 treatment removed eight species; type 3 treatment (filtration) removed four species; and type 6

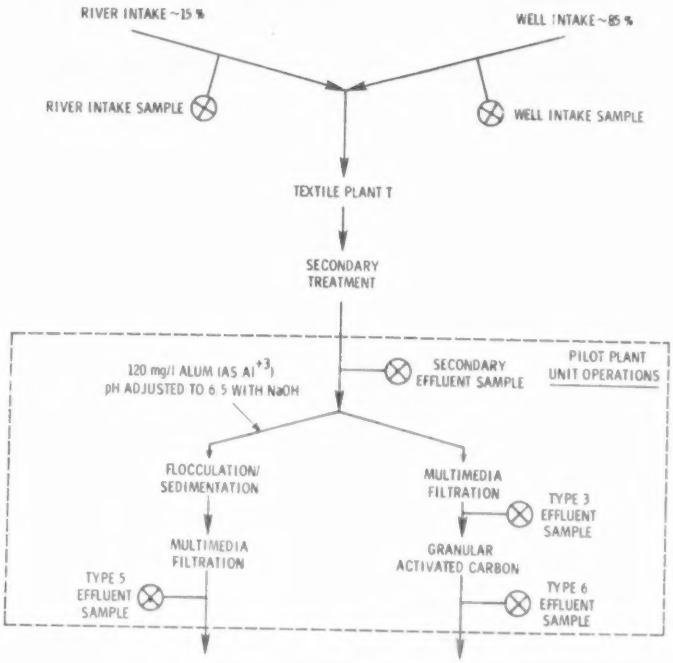
RIVER INTAKE ~15 %
WELL INTAKE ~85 %
RIVER INTAKE SAMPLE
WELL INTAKE SAMPLE
TEXTILE PLANT T
SECONDARY TREATMENT
120 mg/l ALUM (AS Al+3)
pH ADJUSTED TO 6.5 WITH NaOH
SECONDARY EFFLUENT SAMPLE
PILOT PLANT UNIT OPERATIONS
FLOCCULATION/ SEDIMENTATION
MULTIMEDIA FILTRATION
TYPE 3 EFFLUENT SAMPLE
MULTIMEDIA FILTRATION
GRANULAR ACTIVATED CARBON
TYPE 5 EFFLUENT SAMPLE
TYPE 6 EFFLUENT SAMPLE

Figure 17. Candidate wastewater treatment system studied at plant T.

treatment (filtration followed by granular activated carbon) removed six.

Aluminum and sodium were added by type 5 treatment, since alum and NaOH were used in the coagulation step of type 5 treatment. Type 5 treatment removed most of the phosphorus present in plant T secondary effluent.

Daphnia and fathead minnow bioassays provided toxicity data indicating that treatment types 5 (flocculation/sedimentation followed by filtration) and 6 (filtration without coagulation followed by activated carbon) improved the wastewater quality, as shown in Table 45. Treatment type 3 (filtration without precoagulation) slightly improved toxicity to *Daphnia* but had no effect on toxicity to fathead minnows.

TABLE 42. PLANT T ORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, µg/ℓ; percent removal in parentheses)

Pollutant	Well Intake	River Intake	Secondary effluent Phase I	Secondary effluent Phase II	Tertiary effluent Type 3	Tertiary effluent Type 5	Tertiary effluent Type 6
Benzene	7.1	6.2		5.7	6.9	6.8	9.8
Chlorobenzene	–[a]			4.1	4.8	0.1	
1,1-Dichloroethylene				4.2		1.8	1.4
p-Chloro-*m*-cresol					0.6	1.1	
1,1-Dichloroethane							0.5
Ethylbenzene	0.3			0.5	0.2	0.3	
Methylene chloride[b]	24	18		20	19 (5)	18 (10)	19 (5)
Trichlorofluoromethane					0.8		
Phenol		0.7		0.4	1.1	0.3	0.9
Bis(2-ethylhexyl) phthalate	4.8	6.1	23	24	19 (21)	5.2 (78)	14 (42)
Butyl benzyl phthalate	1.2	1.1		5.2	2.5	1.3	
Di-n-butyl phthalate	0.4	0.04		4.4	7.0	1.7	1.7
Tetrachloroethylene			2.9		0.8	1.4	
Toluene	1.2	0.6	33	1.0	0.8	1.0	0.6
Trichloroethylene				0.3	0.4		0.1
Phenol (total)	10	36	41	26	160 (-520)[c]	14 (46)	120 (-360)

[a] Blanks indicate concentration below detection limit (see Table 6).

[b] Methylene chloride may originate from analysis contamination.

[c] Minus percent removals indicate an increase in the concentration of the specified pollutant.

TABLE 43. PLANT T INORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

Pollutant	Well intake	River intake	Secondary effluent Phase I	Secondary effluent Phase II	Tertiary effluent Type 3		Tertiary effluent Type 5		Tertiary effluent Type 6	
Antimony	<10	18[a]	<0.5	54	58	(-7)[b]	49[a]	(9)	39[a]	(28)
Arsenic	<1	<1	<5	3	3		1		3	
Beryllium	<0.04	<0.04	NA[c]	<0.04	<0.04		<0.04		<0.04	
Cadmium	<2	<2	<0.5	2[a]	<2		<2		<2	
Chromium	<4	<4	<0.2	97	95	(2)	20	(79)	84	(13)
Copper	780	8[a]	60	110	100	(9)	18	(84)	87	(21)
Cyanide	<2	<2	<4	11	20	(82)	5	(55)	<2	(>82)
Lead	<22	<22	<1	22[a]	26[a]	(-18)	<22	(>0)	29[a]	(-32)
Mercury	<1	<1	<0.5	<1	<1		<1		<1	
Nickel	55[a]	60[a]	4	93[a]	100[a]	(-8)	59[a]	(37)	90[a]	(3)
Selenium	<1	<1	<5	2	2		2		<1	
Silver	19[a]	27	<5	23[a]	32	(-39)	19[a]	(17)	28[a]	(-22)
Thallium	<50	<50	<5	<50	<50		<50		<50	
Zinc	99	370	80	150	97	(35)	52	(65)	110	(27)

[a] Semiquantitative region; value not within 95% confidence limits.

[b] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[c] Not analyzed.

TABLE 42. PLANT T ORGANIC TOXIC POLLUTANTS DETECTED
(Concentration, μg/ℓ; percent removal in parentheses)

Pollutant	Well Intake	River Intake	Secondary effluent		Tertiary effluent		
			Phase I	Phase II	Type 3	Type 5	Type 6
Benzene	7.1	6.2		5.7	6.9	6.8	9.8
Chlorobenzene	–[a]			4.1	4.8	0.1	
1,1-Dichloroethylene				4.2		1.8	1.4
p-Chloro-*m*-cresol					0.6	1.1	
1,1-Dichloroethane							0.5
Ethylbenzene	0.3			0.5	0.2	0.3	
Methylene chloride[b]	24	18		20	19 (5)	18 (10)	19 (5)
Trichlorofluoromethane					0.8		
Phenol		0.7		0.4	1.1	0.3	0.9
Bis(2-ethylhexyl) phthalate	4.8	6.1	23	24	19 (21)	5.2 (78)	14 (42)
Butyl benzyl phthalate	1.2	1.1		5.2	2.5	1.3	
Di-n-butyl phthalate	0.4	0.04		4.4	7.0	1.7	1.7
Tetrachloroethylene			2.9		0.8	1.4	
Toluene	1.2	0.6	33	1.0	0.8	1.0	0.6
Trichloroethylene				0.3	0.4		0.1
Phenol (total)	10	36	41	26	160 (-520)[c]	14 (46)	120 (-360)

[a] Blanks indicate concentration below detection limit (see Table 6).

[b] Methylene chloride may originate from analysis contamination.

[c] Minus percent removals indicate an increase in the concentration of the specified pollutant.

TABLE 44. PLANT T OTHER POLLUTANTS DETECTED
(Concentration, µg/ℓ; percent removal in parentheses)

Pollutant	Well intake	River intake	Secondary effluent	Tertiary effluent					
				Type 3		Type 5		Type 6	
Aluminum	50[a]	100	160	180	(-13)[b]	3,600	(-2,200)	130	(19)
Barium	5.5	6.0	7.7	7.1		0.6[a]		5.3	
Boron	<1	<1	270	260	(4)	270	(0)	250	(7)
Calcium	13,000	4,700	12,000	11,000	(8)	7,600	(37)	11,000	(8)
Cobalt	<6	<6	<6	<6		<6		<6	
Iron	61	210	520	520	(0)	340	(35)	590	(-13)
Magnesium	4,300	1,500	3,000	3,000	(0)	2,700	(10)	3,300	(-10)
Manganese	300	16	690	710	(-3)	430	(38)	610	(12)
Molybdenum	<10	<10	<10	10[a]		<10		<10	
Sodium	20,000	7,700	180,000	170,000	(6)	370,000	(-106)	170,000	(6)
Phosphorus	140	<70	14,000	13,000	(7)	1,900	(86)	14,000	(0)
Silicon	6,700	2,100	6,400	6,300	(2)	3,300	(48)	6,400	(0)
Tin	<15	<15	<15	<15		<15		<15	
Strontium	85	33	72	70	(3)	50	(31)	70	(3)
Titanium	1.9[a]	3.2[a]	4.0[a]	4.8[a]		4.0[a]		5.0[a]	
Vanadium	33	18	32	35	(-9)	56	(-75)	32	(0)
Ammonia nitrogen	NA[c]	NA	16,000	18,000	(-13)	16,000	(0)	19,000	(-19)
Nitrate nitrogen	NA	NA	1,200	1,200	(0)	1,200	(0)	1,100	(8)
COD	22,000	95,000	630,000	160,000	(75)	140,000	(78)	340,000	(46)
TSS	<1,000	<1,000	20,000	14,000	(30)	28,000	(-40)	12,000	(67)
Sulfide	<3	<3	21	20	(5)	9	(57)	13	(38)
Color[d] at pH 7.6	9	32	177	164	(7)	99	(44)	106	(40)
Color[d] at sample pH	9	26	174	164	(6)	120	(31)	115	(34)
pH[e]	7.6	6.9	8.0	7.6		7.9		7.5	

[a] Semiquantitative region; value not within 95% confidence limits.

[b] Minus percent removals indicate an increase in the concentration of the specified pollutant.

[c] Not analyzed.

[d] ADMI units.

[e] pH units (- log [H$^+$]).

TABLE 45. PLANT T BIOASSAY RESULTS

		Secondary effluent		Tertiary effluent		
Test species	Parameter	Phase I	Phase II	Type 3	Type 5	Type 6
Freshwater algae - *S. capricornutum*	EC_{50} - 7 day, % effluent	NM[a]	>100	>100	>100	>100
	EC_{50} - 12 day, % effluent	NM	>100	>100	>100	>100
	EC_{50} - 14 day, % effluent	-[b]	>100	>100	>100	>100
Daphnia - *D. magna*	LC_{50} - 24 hr, % effluent	NM	54 (47-63)[c]	77 (66-100)	100	>100
	LC_{50} - 48 hr, % effluent	>100[d]	16 (14-19)	23 (23-36)	80 (69-93)	>100
Fathead minnow - *P. promelas*	LC_{50} - 24 hr, % effluent	NM	22 (15-32)	18 (15-22)	68 (46-100)	-[e]
	LC_{50} - 48 hr, % effluent	NM	18 (15-22)	17 (15-19)	56 (46-68)	-[e]
	LC_{50} - 72 hr, % effluent	NM	18 (15-22)	17 (15-19)	56 (46-68)	-[e]
	LC_{50} - 96 hr, % effluent	47	17 (15-19)	17 (15-19)	56 (46-68)	-[e]
S. typhimurium - strains TA98, TA100, TA1535, TA1537, and TA1538	Response to Ames test for mutagenicity - (-) or (+)	(-)	(-)	(-)	(-)	(-)
E. coli - strains W3110 and p3478	Response to pol A test for mutagenicity-increase in zone of inhibition, mm	0	0	0	0	0
Chinese hamster ovary cells	Respone to CHO-K1 test for acute cytotoxicity EC_{50}	-[f]	>100	>100	>100	>100

[a] Not measured.

[b] 20% secondary effluent was extremely stimulatory to the growth of *S. capricornutum*.

[c] 95% confidence limits.

[d] EC_{50} determined with *Daphnia pulex*.

[e] LC_{50} not calculated since mortality data did not follow a normal dose-response relationship; effluent did not appear to be acutely toxic.

[f] EC_{50} not determinable; cytotoxicity procedure employing rabbit alveolar macrophage (RAM) used in Phase I.

TABLE 46. PLANT T EFFLUENT DESCRIPTIONS

Parameter	Secondary effluent		Tertiary effluent		
	Phase I	Phase II	Type 3	Type 5	Type 6
Physical description	Clear, blue green with a moderate amount of particulate, nonchlorinated	Turbid, green-brown liquid	Turbid, dark green liquid	Turbid, light green liquid	Turbid yellow-brown liquid
pH	7.4	7.1	3.5	7.2	7.2
Salinity, g/ℓ	NM[a]	0	0	0	0
Specific conductivity, μmhos/cm²	NM	750	700	1,500	700

[a] Not measured.

SECTION 9

DATA INTERPRETATION

INTRODUCTION

The primary objective of the Phase II textile plant wastewater study was to assess the capabilities of tertiary treatment to remove toxic pollutants and toxicity (as measured by bioassay tests), and to rank the treatment technologies based on this assessment. However, it would be a misuse of the data to accomplish these objectives by a purely quantitative analyses for a number of reasons.

Data collected under the Phase II program are difficult to normalize such that a direct comparison of tertiary treatment system performance can be made.

Concentrations of toxic pollutants and results of bioassay tests varied widely in the secondary effluents and tertiary effluents from a single system type. This was due in part to the semiquantitative nature of the data, but primarily to the fact that treatment studies were conducted at eight different textile mills, each of which discharges a unique wastewater.

Since no more than four tertiary technologies were investigated at a single textile mill, and operating parameters of a single system varied from plant to plant, a reliable basis for comparison of tertiary treatment systems does not lie in the toxic pollutant or bioassay data.

The data does serve, however, to identify preliminary positive and negative results which might be expected in applying various technologies to further treat secondary effluents from textile mills. In most cases, additional research would be required to adequately confirm the preliminary results. In the following discussion, these preliminary results are identified, and supported to the extent possible with data from the Phase II program.

TOXIC ORGANIC REMOVAL CAPABILITIES

Very few toxic organic compounds, other than total phenol, were found in secondary effluents in concentrations greater than 10 μg/ℓ. Bis(2-ethylhexyl) phthalate, methylene chloride, and di-n-butyl phthalate did appear in a few cases, however, the appearance of these compounds could have originated from contaminations by the materials of which the pilot plant was constructed, or by sample analysis techniques. Only data from Plants A and C (see Tables 7 and 12) provide insite into the ability of the tertiary systems to remove other specific toxic organic pollutants. Toluene, 1,2-dichlorobenzene, and 1,2,4-trichlorobenzene were found in concentrations greater than 10 μg/ℓ in the secondary effluent of Plant A. Tertiary system types 2, 5, and 8 were operated at Plant A, and all were effective in reducing the levels of these three compounds. Toluene was found in the secondary effluent of Plant C, and treatment types 2, 5, and 8 again were effective in reducing its concentration. Type 2 treatment of Plant C secondary effluent did appear, however, to add 1,2-dichlorobenzene, although subsequent flitration (type 5 treatment) and activated carbon (type 8 treatment) appeared to remove it.

Total phenol was found in many of the secondary effluents in concentrations greater than 10 μg/ℓ. In both cases in which it was used (Plants A and C), type 8 treatment reduced levels of total phenol. Treatment type 2, used at Plants A, C, and P, treatment type 6, used at Plants W, S, P, N, V, and T, and treatment type 7, used at Plants W and V, appear to be somewhat successful at reducing levels of total phenol. Inconclusive results were obtained when treatment type 3 operated at Plants W, S, P, N, V, and T, treatment type 4, operated at Plants S and V, and treatment type 5, operated at Plants A, C, P, N, and T, were used.

TOXIC METAL REMOVAL CAPABILITIES

Results of toxic metals analysis provided much more insite into the performance of the tertiary systems. Based on the results, treatment types 3, 5, and 6 appear to be the best of the 8 systems at removing toxic metals. Based on data generated at Plants W, S, P, N, and T, treatment type 3 removed antimony in 3 of the 5 cases. In the two remaining cases, the antimony concentration remained about the same. Cadmium was also removed by type 3 treatment in the sole case in which the cadmium concentration in the secondary effluent was greater than 10 μg/ℓ, and type 3 treatment was used (Plant W). Based on data generated at Plants P, N, and T, chromium was removed by type 3 treatment in 2 of the 3 cases. In the third case, its concentration essentially remained the same. At Plants W and P, type 3 was effective in removing copper from secondary effluent, however the copper concentration increased at Plant N. At Plants S, V, and T, the copper concentration remained about the same. Lead was removed by type 3 treatment at Plant W, however, it was added at Plant V. Lead concentrations remained about the same before and after type 3 treatment at Plants S, P, and T. Nickel was removed at Plant T, while concentrations of nickel remained essentially unchanged at Plants S, P, and T. In 5 of 6 cases, zinc was removed by type 3 treatment based on results from Plants W, S, P, N, V, and T. Zinc was added to the secondary effluent at Plant S. In the sole cases in which the metal concentration was greater than 10 µg/ℓ in the secondary effluent, and type 3 treatment was used, arsenic was added based on results at Plant W, and silver was added based on results from Plant T.

As mentioned treatment type 5 also appeared to be effective in removing toxic metals. In 2 of 4 cases, antimony concentrations were reduced by type 5 treatment based upon results from Plants C, P, N, and T. At Plant C, antimony was added, and at Plant T, the antimony concentration remained about the same. Chromium was removed by type 5 treatment in 4 of 5 cases based upon results from Plants A, C, P, N, and T. At Plant c, however, the chromium concentration increased. In 3 of 5 cases, copper was also reduced by type 5 treatment, again based upon results from Plants A, C, P, N, and T. Copper concentrations increased at Plants A and N. Nickel was removed by type 5 treatment in two cases based on results from Plants P and T. Zinc was removed in 3 of 5 cases based upon results from Plants A, C, P, N, and T. At Plant C, zinc appeared to be added, while at Plant A, the zinc concentration remained essentially unchanged Based upon results from a single plant, Plant A, arsenic, beryllium, and cadmium were added to the secondary effluent by type 5 treatment. Lead appears to be added by type 5 treatment based upon results from Plants A, C, P, and T. In two cases, the lead concentration increased, while at the remaining two plants, Plants P and T, secondary and tertiary concentrations were too close to the detection limit of 22 µg/ℓ to draw conclusions from. Type 5 treatment appears to have little effect upon the silver concentration based upon results generated at Plants C and T.

Treatment type 6 also appears to be effective at removing toxic metals. In 4 of 6 cases, antimony was removed by type 6 treatment based on results from Plants W, S, P, N, V, and T. At Plant V, however, antimony appears to be added, and at Plant S, antimony appeared to be uneffected by type 6 treatment. Cadmium appears to be removed by type 6 treatment based upon results from Plant S. Chromium was effectively removed by type 6 treatment at Plants P and N. At Plant T, however, chromium was only slightly removed. In 5 of 6 cases, copper was removed by type 6 treatment based upon results from Plants W, S, P, N, V, and T. At Plant N, the copper concentration increased. Zinc was removed in all cases in which the zinc concentration in the secondary effluent was greater than 10 µg/ℓ, and type 6 treatment was used based upon results from Plants W, S, N, V, and T. Treatment type 6 does not appear to be effective in removing arsenic and silver.

At Plant P, the arsenic concentration increased, while at Plants W and S, arsenic concentrations remained about the same. At Plant W, silver was removed by type 6 treatment, while at Plants V and T, silver concentrations increased as a result of type 6 treatment. Mixed results were obtained when type 6 treatment was used to remove lead and nickel. Lead was removed by type 6 treatment at Plant W, however, it was added at Plant T. The lead concentration appeared to remain essentially the same at Plants S, P, and V. Nickel was removed at Plants W and P, but added at Plant V. At Plants S and T, the nickel concentration before and after type 6 treatment remained about the same.

Treatment types 4 and 7 appeared to be especially ineffective, or detrimental in the removal of toxic metals from secondary effluents. Based on data from Plants S and V, type 4 treatment had essentially no effect upon levels of arsenic, copper, and nickel. At Plant V, antimony, lead, silver, and zinc were added by type 4 treatment. Perhaps, filtration used in type 4 treatment was ineffective in capturing suspended solids formed in the precoagulation step prior to direct filtration. These solids may have contained toxic metals.

Type 7 treatment was used at Plants W and V. Concentrations of antimony, copper, nickel, silver, and zinc increased as a result of the ozonation step used in type 7 treatment both at Plants W and V. In addition, cadmium concentrations increased at Plant W, and chromium increased at Plant V.

TOTAL CYANIDE REMOVAL CAPABILITIES

Treatment types 6 and 7 appear to be the best system of the eight at removing total cyanide whereas treatment type 3 appeared to be the worst. Cyanide was effectively removed by type 6 treatment based on results from Plants W, V, and T where greater than 80% removal efficiencies were observed in all three cases. Cyanide was also effectively removed by type 7 treatment based on results from Plants W and V where greater than 90% removal efficiencies were observed.

On the other hand, cyanide appears to be added by type 3 treatment based on results obtained at Plants W and T.

TOXICITY REMOVAL CAPABILITIES

Toxicity, was measured by bioassay tests, appears to be best removed by treatment types 3 and 6, whereas treatment types 2 and 4 appear to be detrimental to water quality in terms of acute toxicity. In four cases in which acute toxicity to algae was detected in the secondary effluent (Plants W, S, P, and N), type 3 treatment improved water quality in terms of acute toxicity to algae in two cases. At Plant S, acute toxicity to algae remained about the same, and at Plant P, type 3 treatment appeared to be detrimental to water quality. In four cases where acute toxicity to *Daphnia* was detected (Plants W, N, V, and T), type 3 treatment improved water quality in terms of acute toxicity to *Daphnia* in two cases. In the remaining cases, acute toxicity to *Daphnia* remained unchanged. In three cases in which acute toxicity to fathead minnows was detected in the secondary effluent (Plants W, N, and T), water quality in terms of acute toxicity improved in two cases as a result of type 3 treatment, and remained the same in the third case. In the sole case where acute toxicity to bluegills was detected in the secondary effluent (Plant W), no change in acute toxicity was observed as a result of type 3 treatment.

Type 6 treatment also appeared to improve water quality in terms of acute toxicity to various species. In the four cases in which acute toxicity to algae was detected in the secondary effluent, toxicity to algae was removed in three of the cases as a result of type 6 treatment, and in the fourth case, the acute toxicity to algae remained the same. In the cases in which acute toxicity to *Daphnia* was detected in secondary effluents, type 6 treatment removed the toxicity in two cases, and slightly added toxicity in one case. In the three cases in which acute toxicity to fathead minnows was detected in secondary effluent, type 6 treatment removed the toxicity in two of the cases. Toxicity to fathead minnows remained the same in the third case. In the sole case in which acute toxicity to bluegills was detected in the secondary effluent, the toxicity was slightly removed by type 6 treatment.

As mentioned, treatment types 2 and 4 appeared to be detrimental to improving water quality in terms of acute toxicity removal. In two of three cases, toxicity to algae worsened as a result of type 2 treatment based on results from Plants A, C, and P. At Plant A, however acute toxicity to algae only slightly improved. At Plant C, no acute toxicity to *Daphnia* was detected in the secondary effluent, but acute toxicity was detected following type 2 treatment. Toxicity to algae did improve at Plant A as a result of type 2 treatment, however toxicity to bluegills worsened. Based on data from Plants S and V, type 4 treatment added toxicity to algae in both cases, and toxicity to *Daphnia* in one case.

Toxicity increases as a result of treatment types 2 and 4 may have been caused by insufficient removal of residual inorganic coagulant or filter aid used in these treatment systems. For example, the data appear to indicate that tertiary effluents with higher levels of residual aluminum (aluminum present in effluent after discharge from a tertiary treatment system in which alum was used as a coagulant) are more toxic than effluents with lower levels of residual aluminum. Plant A tertiary effluents resulting from type 2, type 5, and type 8 treatment which employ alum and lime coagulation improved in terms of *Daphnia* and algal toxicity, when compared to Plant A secondary effluent. Residual aluminum levels were 1,600 μg/ℓ, 520 μg/ℓ, and 100 μg/ℓ in effluents from type 2 treatment, type 5 treatment, and type 8 treatment, respectively. High levels of residual calcium (70,000 μg/ℓ in all three tertiary effluents, compared to 37,000 μg/ℓ in the secondary effluent) apparently were not high enough to make Plant A tertiary effluents more toxic than Plant A secondary effluent. Similarly, Plant N secondary effluent subjected to type 5 treatment, which employed alum coagulation, improved improved wastewater quality in terms of algal, *Daphnia*, and fathead minnow acute toxicity. Residual aluminum levels in the tertiary effluent were low (110 μg/ℓ aluminum in Plant N secondary effluent and 390 μg/ℓ aluminum in Plant N type 5 effluent).

Conversely, effluents resulting from tertiary treatment of Plant C effluent remained as toxic as, or more toxic than, Plant C secondary effluent with respect to algae, *Daphnia*, and fathead minnows. High levels of residual aluminum were found in type 2, type 5, and type 8 effluents: 13,000 μg/ℓ, 11,000 μg/ℓ, and 9,200 μg/ℓ, respectively. Only 98 μg/ℓ of aluminum was found in Plant C secondary effluent. When a more moderate concentration of aluminum was found in the tertiary effluent (3,600 μg/ℓ aluminum in Plant T effluent treated by the type 5 system), toxicity to algae remained the same as in the secondary effluent, while toxicity to *Daphnia* and fathead minnows improved.

High levels of residual iron or increased chloride levels resulting from ferric chloride coagulation used in type 4 treatment of Plant V effluent may have had a detrimental effect on water quality in terms of algal toxicity. Type 4 effluent was more toxic to algae than Plant V secondary effluent. The residual iron level in the type 4 effluent was 6,200 μg/ℓ, compared to 210 μg/ℓ in Plant V secondary effluent.

The use of cationic polymers may also be detrimental to water quality improvement in terms of acute todicity, especially to algae. In two cases in which polymers were used, toxicity to algae worsened. At Plant S, cationic polymer was used as a filter aid in type 4 treatment, and toxicity to algae worsened significantly. At Plant P, cationic polymer was used as a coagulant in type 2 treatment, and toxicity to algae worsened significantly. After flocculation/sedimentation (type 2 treatment) at Plant P, the wastewater was treated with carbon (type 5 treatment). As a result, acute toxicity to algae improved with respect to that observed with the type 2 effluent, however, the acute toxicity of the type 5 effluent was still worse than that observed with the secondary effluent.

OTHER OBSERVATIONS

In analyzing bioassay data generated in the Phase II program, several other observations can be made. Generally, freshwater algae toxicity testing was the most sensitive of the bioassays employed in the Phase II study. Toxicity to algae (EC_{50} - 14 days less than 100%) was detected in 74% of the samples tested; toxicity to *Daphnia* (LC_{50} - 48 hr less than 100%) was detected in 54% of the samples tested; toxicity to bluegills (LC_{50} - 96 hr less than 100%) was detected in 44% of the samples tested; and toxicity to fathead minnows (LC_{50} - 96 hr, less than 100%) detected in 33% of the samples tested. No toxicity was detected when any of the microbiological bioassay techniques were used.

Overall, the secondary effluents at the eight textile plant locations subjected to MRC sampling/analysis/bioassay during both Phase I and Phase II have improved in terms of toxicity, since Phase I testing. An improvement was observed at five plants, while degradation of secondary effluent quality in terms of toxicity was observed at one plant.

OVERVIEW

In summary, a number of trends concerning toxic pollutant levels, toxicity levels, and tertiary treatment type have been identified by analyzing the Phase II data. These trends are described below.

1. Multimedia filtration without precoagulation followed by granular activated carbon (type 6 treatment) was the best **system** employed in terms of removing both toxicity and toxic pollutants. Other tertiary systems rank as follows:

	Removal of toxic organic compounds	Removal of toxic metals	Removal of cyanide	Removal of acute toxicity	Composite
Best removal ability	-[a]	Type 3, Type 5, Type 6	Type 6, Type 7	Type 3, Type 6	Type 6
Intermediate removal ability		Type 1, Type 2, Type 8	Type 1, Type 2, Type 4, Type 5, Type 8	Type 1, Type 5, Type 7, Type 8	Type 1, Type 2, Type 3, Type 5, Type 7, Type 8
Least removal ability		Type 4, Type 7	Type 3	Type 2, Type 4	Type 4

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[a] Inadequate data with which to base conclusions.

2. Systems employing alum or iron coagulation, in which residual concentrations of alum or iron were high (greater than 9,000 μg/ℓ Al or greater than 6,000 μg/ℓ Fe), generally increased the toxicity of the wastewater.

3. Coagulation with cationic polymers appeared to be detrimental to freshwater algae.

4. Freshwater algae toxicity testing was the most sensitive of the bioassays employed in the Phase II study.

5. Overall, the secondary effluents at the eight textile plant locations subjected to MRC sampling/analysis/bioassay during both Phase I and Phase II have improved in terms of toxicity, since Phase I testing.

SECTION 10

RESULTS OF SPECTRA ANALYSIS OF PHASE I SAMPLES

TOTAL ORGANIC CONCENTRATION

Another portion of the Phase I chemical characterization study involved determining the total organic concentration (4). Following the procedure specified by EPA, 10 ℓ of each secondary effluent sample were filtered through 0.45-μm filter paper (12). A portion of this sample was then extracted three times with methylene chloride. The extracts were then dried at 105°C and weighed to determine the total amount of methylene chloride-extractable, nonvolatile organics. Results of this analysis are shown in Table 47 (12).

TABLE 47. CONCENTRATION OF METHYLENE CHLORIDE-EXTRACTABLE ORGANICS IN FILTERED SECONDARY EFFLUENTS

Plant	Organic concentration, g/m^3	Plant	Organic concentration, g/m^3
A	63.7	N	9.24
B	3.18	S	5.40
C	28.2	T	17.8
E	3.60	U	14.6
F	16.0	V	–[a]
G	27.2	W	15.0
K	2.73	X	13.5
L	18.3		

[a] Analysis not performed.

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(12) Hamersma, J. W., S. L. Reynolds, and R. F. Maddalone. IERL-RTP Procedures Manual: Level 1 Environmental Assessment. EPA-600/2-76-106a (PB 257 850), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, June 1976. 147 pp.

PHASE I PRIORITY POLLUTANTS

For the purposes of data comparison, Table 48 shows the organic priority pollutants found in the 23 secondary effluents in Phase I of the study (4).

OTHER ORGANIC COMPOUNDS IDENTIFIED

Results of re-examining the GC/MS spectra of the Phase I secondary effluent samples are shown in Tables 49 through 71. Each table shows the results from 1 of the 23 samples, and each is divided to show which compounds were detected in each of the three priority pollutant fractions: volatile, base/neutral, and acid fraction organics.

Methylene chloride was found in all volatile samples, but its presence is principally due to laboratory contamination. Some of the data stored on magnetic tape were lost due to a power surge, and some data were not retrievable due to GC/MS scanning malfunctions.

TABLE 48. PHASE I PRIORITY POLLUTANTS IN SECONDARY EFFLUENTS

Plant	Compound identified and concentration observed, µg/ℓ		
	Volatiles	Base/neutrals	Acids
A	Toluene 8.4	1,2-Dichlorobenzene 1 1,4-Dichlorobenzene 0.05 Bis(2-ethylhexyl) phthalate 6 1,2,4-Trichlorobenzene 46	NPPO[a]
B	Trichlorofluoromethane 2.6	N-nitroso-di-n-propylamine 2 Bis(2-ethylhexyl) phthalate 3 Pyrene 0.3	NPPO
C	Toluene 2.6 Ethylbenzene 2	1,2-Dichlorobenzene 0.3 1,2,4-Trichlorobenzene 10 Acenaphthene 0.5 Bis(2-ethylhexyl) phthalate 3 Anthracene 4.4	NPPO
D	Toluene 1.7	Diethyl phthalate 1 Bis(2-ethylhexyl) phthalate 5	NPPO
E	Toluene 5.5	1,2-Dichlorobenzene 0.2 1,4-Dichlorobenzene 0.2 Dimethyl phthalate 1 Diethyl phthalate 0.5 Bis(2-ethylhexyl) phthalate 18 Pyrene 0.1	NPPO
F	Trichlorofluoromethane 1.7 *Trans*-1,3-dichloropropene 3.9 *Cis*-1,3-dichloropropene 5.6 Toluene 0.8 Ethylbenzene 2.7	1,2,4-Trichlorobenzene 6.3 Bis(2-ethylhexyl) phthalate 23	2,4-Dimethylphenol 9
G	Toluene 0.8	Acenaphthene 2 Hexachlorobenzene 0.8 Diethyl phthalate 11 Bis(2-ethylhexyl) phthalate 10	Phenol 2
H	Toluene 12 Trichlorofluoromethane 2,100	Bis(2-ethylhexyl) phthalate 230	NPPO
J	Toluene 8 Ethylbenzene 51	Bis(2-ethylhexyl) phthalate 35 Di-n-butyl phthalate 3.6 Pyrene 0.1	NPPO
K	Chloroform 58 Trichloroethylene 4.6 Toluene 24 Ethylbenzene 0.7	Naphthalene 0.5 Bis(2-ethylhexyl) phthalate 8	NPPO
L	Benzene 0.5	Bis(2-ethylhexyl) phthalate 2	NPPO
M	Toluene 0.4	1,2,4-Trichlorobenzene 1.8 Di-n-butyl phthalate 58	NPPO

(continued)

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[a] No priority pollutants observed.

TABLE 48 (continued).

	Compound identified and concentration observed, μg/ℓ		
Plant	Volatiles	Base/neutrals	Acids
N	Toluene 17 Ethylbenzene 75	1,2-Dichlorobenzene 6 1,4-Dichlorobenzene 1.5 Diethyl phthalate 9.4 Bis(2-ethylhexyl) phthalate 16.7	2,4-Dimethylphenol 8
P	Chloroform 6.9 Toluene 22 Ethylbenzene 280	Bis(2-ethylhexyl) phthalate 72 N-nitroso-di-n-propylamine 19	NPPO[a]
R	Toluene 17 Ethylbenzene 29	Diethyl phthalate 2 Bis(2-ethylhexyl) phthalate 12	Chloro cresol 32 Pentachlorophenol 56
S	Toluene 21 Ethylbenzene 110 1,1,2,2-Tetrachloroethylene 0.4	Bis(2-ethylhexyl) phthalate 41 Naphthalene 255 1,2,4-Trichlorobenzene 916	NPPO
T	Toluene 33 1,1,2,2-Tetrachloroethylene 3	Bis(2-ethylhexyl) phthalate 23	NPPO
U	Chloroform 18 Bromodichloromethane 1.5 *Trans*-1,3-dichloropropene 0.9 Toluene 13	Bis(2-ethylhexyl) phthalate 140 Naphthalene 22	NPPO
V	Toluene 1,400	Bis(2-ethylhexyl) phthalate 9	NPPO
W	Toluene 1.7	Bis(2-ethylhexyl) phthalate 19	NPPO
X	Toluene 40 Trichlorofluoromethane 35 1,1,2,2-Tetrachloroethylene 41	Hexachlorobenzene 0.5 Diethyl phthalate 3 Bis(2-ethylhexyl) phthalate 2	
Y	Chloroform 5 Trichlorofluoromethane 10	1,2-Dichlorobenzene 0.6 Naphthalene 0.6 Diethyl phthalate 3 Di-n-butyl phthalate 7 Hexachlorobenzene 0.3 Bis(2-ethylhexyl) phthalate 25	NPPO
Z	Toluene 110 Ethylbenzene 3,020 Chlorobenzene 3.5 Trichlorofluoromethane 89	Bis(2-ethylhexyl) phthalate 2	NPPO

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[a] No priority pollutants observed.

TABLE 49. PLANT A: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Bromochloromethane	Minor
Dichlorobenzene	Minor
1 unknown (possibly an alkyl alcohol)	Minor
Base/neutrals	
Dichlorobenzene	0.1
Trichlorobenzene	4.2
Aliphatics (C_{15} on up, paraffinic)	52.0
Aliphatics (C_{15} on up, olefinic)	43.6
Di-C_8 alkyl phthalate	0.1
Acids	
C_8-aliphatic acid	0.1
Trichlorobenzene	0.1
C_9-aliphatic acid	0.1
C_{10}-aliphatic acid	0.1
C_{12}-aliphatic acid	0.2
C_{14}-aliphatic acid	0.4
Aliphatics (olefins C_{12} and up)	95.3
C_{16}-aliphatic acid	1.2
C_{18}-aliphatic acid	1.5
C_{20}-aliphatic acid	1.0

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[a] Methylene chloride presence due to laboratory contamination.

TABLE 50. PLANT B: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Bromodichloromethane	Minor
Base/neutrals	
Aliphatics (C_{16}-C_{32})	44.1
Diethyl phthalate	0.9
Dipropyl phthalate	1.8
Dibutyl phthalate	1.0
Di-C_8 alkyl phthalate	1.0
Triphenylphosphine	7.7
Triphenylphosphine oxide	9.6
Triphenylphosphine sulfide	2.1
C_{13}-hydroxy amide	0.6
C_{15}-hydroxy amide	6.0
C_{17}-hydroxy amide	13.7
Unknown ester, $C_{21}H_{34}O_2$	0.4
Unknown ester, $C_{21}H_{34}O_2$	0.6
Unknown ester, not a methyl ester ($\sim C_{17}$ or C_{18})	4.5
Unknown ester, possibly methyl dehydroabietate	0.8
Compound, either dihydroxyacetophenone or acetylhydroquinone	2.0
C_9-alkyl phenols	1.2
Anthracene/phenanthrene	0.7
Methyl-anthracenes/-phenanthrenes	0.4
Dimethyl-anthracenes/-phenanthrenes	0.3
Fluoranthene	0.1
Pyrene	0.4
Methyl-fluoranthenes/-pyrenes	0.1
Acids	–[b]
C_{10}-aliphatic	
C_{11}-aliphatic	
C_{12}-aliphatic	
C_8-aliphatic acid	
C_{13}-aliphatic	
C_9-aliphatic acid	
C_{10}-aliphatic acid	
C_{15}-aliphatic	
C_{12}-aliphatic acid (lauric acid)	
C_{16}-aliphatic	
C_{14}-aliphatic acid	
C_{15}-aliphatic acid	
C_{16}-aliphatic acid	
C_{17}-aliphatic acid	
C_{18}-aliphatic acid	

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[a] Methylene chloride presence due to laboratory contamination.

[b] In general, C_{10}-C_{16} aliphatics are weak (~7% based on area for 43 ion). Among C_8-C_{18} aliphatic acids, C_{12}-C_{14} are moderate and C_{16} and C_{18} are strong (total acids ~93% based on 73 and 60 ions).

TABLE 51. PLANT C: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Bromodichloromethane	Minor
Toluene	Minor
Base/neutrals	
Data not retrievable from magnetic tape	
Acids	
Alcoholic ether, unknown	7.3
Butoxy ethanol	5.3
Phenol	0.8
Diethylene glycol ether	4.0
Cresols	0.3
Dimethylphenols	0.6
Benzoic acid	1.7
C_{10}-aliphatic acid	1.8
C_{12}-aliphatic acid	5.6
C_{14}-aliphatic acid	12.2
C_{16}-aliphatic acid	24.8
C_{18}-aliphatic acid	35.6

[a] Methylene chloride presence due to laboratory contamination.

TABLE 52. PLANT D: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Data not retrievable from magnetic tape	
Base/neutrals	
Aliphatics (C_{16}→on up, primarily paraffinic)	92.2
Unknown ester, $\sim C_{16}$, not a methyl ester	2.3
Diethyl phthalate	0.6
Dibutyl phthalate	2.9
Di-C_8 alkyl phthalate	1.2
Benzanthrene	0.8
Acids	
Butoxy ethanol	2.0
Diethylene glycol ether	3.0
Unknown aliphatic ether/alcohol	2.0
Benzoic acid	7.5
C_{15}-aliphatic	2.0
Undeterminated phthalate	5.5
C_{14}→C_{17}-aliphatic acids (& C_{18}-above)	78.0

TABLE 53. PLANT E: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Carbon dioxide	Minor
Methylene chloride[a]	Major
Toluene	Minor
Xylenes	Minor
Ethylbenzenes	Minor
Base/neutrals	
Dichlorobenzenes	0.2
Naphthalene	0.1
Methyl-naphthalenes	<0.1
Biphenyl	<0.1
Phthalic acid/or anhydride	0.7
Unknown ester	11.4
Dichlorodimethoxybenzene	0.3
Unknown, possibly dichloro-trimethoxybenzene	0.1
Diethyl phthalate	0.4
C_{15}-hydroxy amide	2.9
C_{17}-hydroxy amide	7.1
Dibutyl phthalate	0.5
Di-C_8-alkyl phthalate	5.4
Triphenylphosphine	3.1
Triphenylphosphine oxide	1.8
Triphenylphosphine sulfide	0.5
Aliphatics (C_{15}→on up, paraffinic character)	40.3
Aliphatics (C_{15}→on up, olefinic character)	25.2
Acids	
Butoxy ethanol	1.1
Phenol	1.5
Cresols	0.5
Dimethylphenols	1.9
C_8-aliphatic acid	0.6
C_{10}-aliphatic acid	0.7
Tetrachlorophenol	0.4
Pentachlorophenol	3.9
Di-(C_8-alkyl) phthalate	9.5
C_{12} & above aliphatic acids	16.2
C_{16} & above aliphatics	63.7

[a] Methylene chloride presence due to laboratory contamination.

TABLE 54. PLANT F: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Only peak
Base/neutrals	
Alkyl oxygenates (e.g., ethers), dominant 59 ion	0.8
Alkyl oxygenates (e.g., ethers), dominant 45 ion	0.7
Unknown (2) with dominant 42 & 45 ions	6.7
Alkyl oxygenates (e.g., ethers), dominant 59 ion	14.3
Aliphatics, C_{15} & above	68.9
Trichlorobenzenes	0.5
C_9-alkylphenols	1.2
Diethyl phthalate	0.1
Dibutyl phthalate	0.2
Di-C_8 alkyl phthalate	4.8
Triphenyl phosphine	0.2
Triphenylphosphine oxide	0.8
Triphenylphosphine sulfide	0.8
Acids	
Phenol	0.5
Methyl benzoate	0.1
Benzoic acid	21.7
Toluic acid	14.5
C_{12}-methyl ester	1.8
C_{13}-methyl ester	0.4
C_{16}-methyl ester (palmitate)	0.8
C_{17}-methyl ester	0.2
C_{18}-methyl ester (stearate)	2.3
Biphenyl	0.7
C_{10} & above aliphatic acids	10.5
C_{15} & above aliphatics	46.5

[a] Methylene chloride presence due to laboratory contamination.

TABLE 55. PLANT G: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Toluene	Minor
C_3-alkyl benzene	Minor
Base/neutrals	
Aliphatics (C_{16} & up), paraffinic character	54.5
Aliphatics (C_{16} & up), olefinic character	45.3
Dibutyl phthalate	0.1
Di-C_8 alkyl phthalate	0.1
Triphenylphosphine	<0.1
Triphenylphosphine oxide	<0.1
Triphenylphosphine sulfide	<0.1
Acids	
Butoxyethanol	4.6
C_3-alkyl benzene	0.1
C_8-aliphatic acid	0.4
C_9-aliphatic acid	0.2
Naphthalene	1.1
C_{10}-aliphatic acid	0.5
C_{12}-aliphatic acid	5.6
Caprolactam	6.4
Biphenyl	3.2
C_{14}-aliphatic acid	2.3
C_{15}-aliphatic acid	2.6
C_{18}-aliphatic acid	2.6
C_{16} & above aliphatics	70.4

[a] Methylene chloride presence due to laboratory contamination.

TABLE 56. PLANT H: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Carbon dioxide	Minor
Methylene chloride[a]	Major
Acetone	Minor
Trichlorofluoromethane	Minor
Unknown (possibly an alkyl alcohol)	Minor
Methyl pentanol	Minor
Hexane	
Toluene	
Base/neutrals	
Unknown (45 ion), similar to ethoxy ethyl acetate	2.9
Aliphatics, $C_{15}\rightarrow$on up (paraffinic character)	54.1
Aliphatics, $C_{15}\rightarrow$on up (olefinic character)	39.5
Di-t-butylphenol	0.1
Unknown, ester, C_{15} or C_{16}	1.6
Diethyl phthalate	0.2
Dibutyl phthalate	0.2
Di-C_8 alkyl phthalate	1.3
Ester, $C_{21}H_{34}O_2$	0.05
Ester, $C_{21}H_{34}O_2$	0.05
Acids	
Weak alcoholic ethers	Very weak
C_{12} & above aliphatics	Very weak

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[a] Methylene chloride presence due to laboratory contamination.

TABLE 57. PLANT J: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Toluene	Minor
Ethylbenzene	Major
Base/neutrals	
Oxindole	0.1
Toluidine/or methyl aniline	0.2
C_8-alkylphenol	0.3
C_9-alkylphenols	1.3
Unknown ester, dominant 71 ion	1.4
Unknown esters (3), dominant 57 ion (possibly long-chain alkyl esters of acids $< C_5$)	2.0 2.9 4.8
Aliphatics, C_{20} & above (paraffinic character)	56.9
Aliphatics, C_{20} & above (olefinic character)	27.1
Acids	
C_8 & above aliphatics	Very weak

[a] Methylene chloride presence due to laboratory contamination.

TABLE 58. PLANT K: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Bromochloromethane	Minor
Chloroform	Minor
Trichloroethylene	Minor
Base/neutrals	
Diiodochloromethane	1.1
Aliphatics (C_{15}-on up, paraffinic character)	57.4
Aliphatics (C_{15}-on up, olefinic character)	26.4
Unknown ester, C_{15} or C_{16}	5.5
Diethyl phthalate	0.9
Anthracene/phenanthrene	0.5
Dipropyl phthalate	0.2
Methyl-anthracenes/-phenanthrenes	0.2
Dibutyl phthalate	0.6
C_{15}-hydroxy amide	0.7
Dimethyl-anthracenes/-phenanthrenes	0.1
Fluoranthene	0.05
Pyrene	0.05
Ester, $C_{21}H_{34}O_2$	0.3
Dioctyl adipate	0.1
C_{17}-hydroxy amide	1.4
Triphenylphosphine	1.1
Ester, $C_{21}H_{34}O_2$	0.6
Triphenylphosphine oxide	1.2
Triphenylphosphine sulfide	0.8
Di-C_8 alkyl-phthalate	0.8
Acids	
Diethylene glycol ether	15.5
Unknown (alcoholic ether/or di-ether)	1.3
Aliphatic (C_9-olefin)	0.6
Benzoic acid	1.1
C_{18}-aliphatic acid	0.2
C_{12}-aliphatic acid	1.7
Trichlorophenol	0.1
C_{14}-aliphatic acid	3.8
C_{18}-aliphatic acid	25.9
C_{18}-aliphatic acid	28.7
Pentachlorophenol	0.3
Di-C_8-alkyl-adipate	20.8

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[a] Methylene chloride presence due to laboratory contamination.

TABLE 59. PLANT L: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Base/neutrals	
Data not retrievable from magnetic tape	
Acids	
Dimethylformamide	23.1
Dimethylacetamide	24.0
Unknown	1.8
Cresols	1.0
Alcoholic ether	11.2
C_{12}-aliphatic acid	4.1
Unknown-possibly ethyl hydrogen phthalate	5.0
C_{14}-aliphatic acid	3.5
C_{16}-aliphatic acid	11.1
C_{18}-aliphatic acid	15.2

[a] Methylene chloride presence due to laboratory contamination.

TABLE 60. PLANT M: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Only peak
Base/neutrals	
Data not retrievable from magnetic tapes	
Acids	
Unknown	1.3
Phenol	4.9
Diethylene glycol ether	5.9
Nonlinear C_8-aliphatic acid	1.4
Cresols	0.1
C_8-aliphatic acid	1.4
C_9-olefin or cyclic alkane	1.0
Benzoic acid	2.0
Dimethylphenols	0.1
Unknown alcoholic ether	6.1
C_{10}-aliphatic acid	3.6
C_{15}-aliphatic	1.4
C_{12}-aliphatic acid	8.4
Trichlorophenol	0.1
C_{16}-aliphatic	1.5
C_{14}-aliphatic acid	9.6
C_{16}-aliphatic acid	22.8
C_{18}-aliphatic acid	28.4
C_{20}-aliphatic acid	28.4

[a] Methylene chloride presence due to laboratory contamination.

TABLE 61. PLANT N: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Bromochloromethane	Minor
Unknown (possibly an alkyl alcohol)	Minor
C_2-alkyl benzene	Minor
Dichlorobenzene	Minor
Base/neutrals	
Dichlorobenzenes	0.4
Dichlorodimethoxybenzenes	<0.1
Dichloroaniline	2.6
C_{10}-C_{14} alkyl benzenes	11.9
C_8-alkyl phenol	0.4
C_9-alkyl phenols	3.8
Di-C_8 alkyl phthalate	0.1
Aliphatics, C_{15}→on up, primarily paraffinic	80.8
Acids	
Data not retrievable from magnetic tapes	

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[a] Methylene chloride presence due to laboratory chloride.

TABLE 62. PLANT P: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Minor
Chloroform	Minor
Toluene	Minor
C_2-alkyl benzene	Major
Base/neutrals	
Benzaldehyde	0.4
Benzoyl alcohol	47.7
Aliphatics, C_{14}-C_{24}	16.6
Unknown ester ($\sim C_{15}$ or C_{16})	3.2
Unknown (192/190/188 isotope)	0.3
Diethyl phthalate	0.4
Anthracene/phenanthrene	0.1
Methyl-anthracenes/-phenanthrenes	0.1
Dimethyl-anthracenes/-phenanthrenes	0.1
Fluoranthene	0.05
Pyrene	0.05
Dibutyl phthalate	0.6
Ester $C_{21}H_{34}O_2$ (MW 318)	2.2
Ester $C_{21}H_{34}O_2$ (MW 318)	0.6
Dioctyl adipate	14.3
Unknown (94 ion)	1.7
Di-C_8 alkyl-phthalate	7.7
Unknown (70 ion)-possibly an ester	0.3
Long-chain alkyl esters (C_{22} and greater)	3.6
Acids	
C_2-alkylbenzenes (ethyl benzene/ xylenes)	0.8
C_{10}-aliphatic	2.2
C_{11}-aliphatic	5.0
Benzoyl alcohol	63.8
Benzoic acid	0.4
C_{12}-aliphatic acid	1.0
Ethyl hydrogen phthalate	1.0
C_{14}-aliphatic acid	0.9
C_{13}-hydroxy amide	1.1
C_{16}-aliphatic acid	1.9
C_{15}-hydroxy amide	7.8
C_{18}-aliphatic acid	2.7
C_{17}-hydroxy amide	11.4

[a] Methylene chloride presence due to laboratory contamination.

TABLE 63. PLANT R: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Only peak
Base/neutrals	
Data not retrievable from magnetic tapes	
Acids	
Data not retrievable from magnetic tapes	

[a] Methylene chloride presence due to laboratory contamination.

TABLE 64. PLANT S: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Acetone	Minor
Bromochloromethane	Minor
Toluene	Minor
C_2-alkyl benzene	Minor
Base/neutrals	
C_3-alkyl benzenes	2.2
C_4-alkyl benzenes	2.5
C_5-alkyl benzenes	1.5
Aliphatics, C_8 & above (paraffinic character)	54.5
Aliphatics, C_8 & above (olefinic character)	32.8
Butyl benzoate	1.7
Trichlorobenzenes	1.0
Dimethyl aniline	1.5
Anisidine	0.8
Naphthalene	0.6
Methyl-naphthalenes	0.3
Biphenyl	0.5
Di-C_8 alkyl-phthalate	0.1
Acids	
Dimethyl acetamide	0.6
Benzoic acid	3.2
C_{12}-aliphatic acid	2.0
C_{14}-aliphatic acid	3.5
C_9 & above aliphatics	78.6
C_{16}-aliphatic acid	5.8
C_{18}-aliphatic acid	6.3

[a] Methylene chloride presence due to laboratory contamination.

TABLE 65. PLANT T: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Acetone	Minor
Bromochloromethane	Minor
Tetrachloroethylene	Minor
Base/neutrals	
Aliphatics (C_{13} on up, mixed species)	58.9
Alkyl oxygenates (e.g., ethers), dominant 59 ion	16.7
Alkyl oxygenates (e.g., ethers), dominant 45 ion	13.4
Unknown, isotopic mass 195, containing Cl, N, S	<0.1
C_8-alkylphenol	5.5
C_9-alkylphenols	5.2
Di-C_8 alkyl phthalate	0.3
Acids	
Data not retrievable from magnetic tapes	

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[a] Methylene chloride presence due to laboratory contamination.

TABLE 66. PLANT U: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Bromodichloromethane	Minor
Chloroform	Minor
Toluene	Minor
Base/neutrals	
Chloroaniline	0.3
Aliphatics (C_{12}-on up, paraffinic character)	62.5
Aliphatics (C_{12}-on up, olefinic character)	36.2
C_6 and C_7 alkyl-phthalates	0.7
Di-C_8 alkyl-phthalate	0.3
Triphenylphosphine oxide	<0.1
Triphenylphosphine sulfide	<0.1
Acids	
Butoxy ethanol	4.5
Phenol	0.3
C_6-alkene	4.6
o-Methoxyphenol	0.2
Benzoic acid	3.7
C_{10}-aliphatic acid	0.4
Benzothiazole	4.0
C_{12}-aliphatic acid	2.2
C_{14}-aliphatic acid	4.4
C_{16}-aliphatic acid	23.7
C_{18}-aliphatic acid	35.8
Di-C_8 alkyl-phthalate	16.2

[a] Methylene chloride presence due to laboratory contamination.

TABLE 67. PLANT V: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Toluene	Minor
Base/neutrals	
Aliphatics, C_{16} & above (primarily paraffinic)	98.8
Methylthiobenzthiozole	0.2
Dibutyl phthalate	0.5
Di-C_8 alkyl phthalate	0.2
Triphenylphosphine oxide	0.2
Triphenylphosphine sulfide	0.1
Acids	
Data not retrievable from magnetic tapes	

[a] Methylene chloride presence due to laboratory contamination.

TABLE 68. PLANT W: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Only peak
Base/neutrals	
Phenol	0.1
Benzaldehyde	0.1
3 Unknowns, dominant 100 ion	1.8
$C_7 \rightarrow C_9$ alkylphenols	13.1
Dipropyl phthalate	0.5
Dibutyl phthalate	0.2
Di-C_8 alkyl phthalate	0.9
Unknown ester, dominant 71 ion	1.4
Aliphatics, C_{15} & above	81.9
Acids	
Data not retrievable from magnetic tapes	

[a] Methylene chloride presence due to laboratory contamination.

TABLE 69. PLANT X: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Acetone	Minor
Bromochloromethane	Minor
Trichloroethylene	Minor
Tetrachloroethylene	Minor
Unknown (possibly an alkyl alcohol)	Weak
C_3-alkyl benzene	Weak
Base/neutrals	
Data not retrievable from magnetic tapes	
Acids	
Tetrachloroethylene	0.05
Butoxy ethanol	0.7
Gamma butyrolacetone	1.8
Diethylene glycol ether	2.8
N-methyl-2-pyrrolidine	1.9
Benzoic acid	2.4
Trichlorobenzene	0.4
Naphthalene	0.05
C_{10}-aliphatic	0.3
Methyl naphthalenes	0.2
Isobutyl benzoate	1.0
Caprolactam	0.8
Biphenyl	1.7
Vanillin	0.4
Phenylphenol	2.0
C_{14}-C_{17}-aliphatic acids	10.9
C_{16}-C_{18}-aliphatics	50.1
Di-(C_8-alkyl)-adipate	22.5

---

[a] Methylene chloride presence due to laboratory contamination.

TABLE 70. PLANT Y: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Acetone	Minor
Trichloroethylene	Minor
Base/neutrals	
Dichlorobenzenes (2 isomers)	0.5
Toluidine	0.4
C_4-alkylphenol	0.2
Chlorotoluidine	0.4
Dichloroaniline	0.4
Di-t-butylphenol	0.5
Aliphatics, $C_{15}\rightarrow$on up (paraffinic character)	63.0
Aliphatics, $C_{15}\rightarrow$on up (olefinic character)	28.1
Unknown ester, C_{15} or C_{16}	3.6
Azobenzene	0.4
Phenanthrene/anthracene	0.2
Diethyl phthalate	0.4
Dibutyl phthalate	0.2
Di-C_8 alkyl-phthalate	1.3
Benzanthrone	0.4
Acids	
Phenol	1.2
2-Ethyl hexanoic acid	0.7
Benzoic acid	69.5
Nitrocresol or nitroanisole	7.8
$C_{12}\rightarrow C_{18}$ aliphatic acids	20.8

[a] Methylene chloride presence due to laboratory contamination.

TABLE 71. PLANT Z: OTHER GC/MS ORGANIC COMPOUNDS IN SECONDARY EFFLUENT

Compounds	Estimated percent
Volatiles	
Methylene chloride[a]	Major
Trichlorofluoromethane	Minor
Hexane	Minor
Toluene	Minor
C_2-alkyl benzene	Minor
Base/neutrals	
Data not retrievable from magnetic tapes	
Acids	
Data not retrievable from magnetic tapes	

[a] Methylene chloride presence due to laboratory contamination.

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APPENDIX

TOXIC POLLUTANT ANALYSIS FRACTIONS

TABLE A-1. VOLATILE AND DIRECT INJECTABLE COMPOUNDS

Compound
Chloromethane
Dichlorodifluoromethane
Bromomethane
Vinyl chloride
Chloroethane
Methylene chloride
Trichlorofluoromethane
1,1-Dichloroethylene
1,1-Dichloroethane
Trans-1,2-dichloroethane
Chloroform
1,2-Dichloroethane
1,1,1-Trichloroethane
Carbon tetrachloride
Bromodichloromethane
Bis(chloromethyl) ether
1,2-Dichloropropane
Trans-1,3-dichloropropene
Trichloroethylene
Dibromochloromethane
Cis-1,3-dichloropropene
1,1,2-Trichloroethane
Benzene
2-Chloroethyl vinyl ether
Bromoform
1,1,2,2-Tetrachloroethylene
1,1,2,2-Tetrachloroethane
Toluene
Chlorobenzene
Ethylbenzene
Acrolein
Acrylonitrile

TABLE A-2. BASE NEUTRAL EXTRACTABLE COMPOUNDS

Compound	Compound
1,3-Dichlorobenzene	Anthracene
1,4-Dichlorobenzene	Diethyl phthalate
Hexachloroethane	Dimethyl phthalate
1,2-Dichlorobenzene	Fluoranthene
Bis(2-chloroisopropyl) ether	Pyrene
Hexachlorobutadiene	Di-n-butyl phthalate
1,2,4-Trichlorobenzene	Benzidine
Naphthalene	Butyl benzyl phthalate
Bis(2-chloroethyl) ether	Chrysene
Hexachlorocyclopentadiene	Bis(2-ethylhexyl) phthalate
Nitrobenzene	Benzo(a)anthracene
Bis(2-chloroethoxy) methane	Benzo(b)fluoranthene
2-Chloronaphthalene	Benzo(k)fluoranthene
Acenaphthylene	Benzo(a)pyrene
Acenaphthene	Indeno(1,2,3-cd)pyrene
Isophorone	Dibenzo(a,h)anthracene
Fluorene	Benzo(g,h,i)perylene
2,6-Dinitrotoluene	N-nitrosodimethylamine
1,2-Diphenylhydrazine	N-nitroso-di-n-propylamine
2,4-Dinitrotoluene	4-Chlorophenyl phenyl ether
N-nitrosodiphenylamine	3,3'-Dichlorobenzidine
Hexachlorobenzene	2,3,7,8-Tetrachlorodibenzo-*p*-dioxin[a]
4-Bromophenyl phenyl ether	
Phenanthrene	Bis(chloromethyl) ether

[a] This compound was specifically listed in the consent decree. Because of TCDD's extreme toxicity, EPA recommends that laboratories not acquire analytical standards for this compound.

TABLE A-3. ACID EXTRACTABLE COMPOUNDS

2-Chlorophenol
Phenol
2,4-Dichlorophenol
2-Nitrophenol
p-Chloro-*m*-cresol
2,4,6-Trichlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
4,6-Dinitro-*o*-cresol
4-Nitrophenol
Pentachlorophenol

TABLE A-4. PESTICIDES AND PCB's

Compound
β-Endosulfan
α-BHC
γ-BHC
β-BHC
Aldrin
Heptachlor
Heptachlor epoxide
α-Endosulfan
Dieldrin
4,4'-DDE
4,4'-DDD
4,4'-DDT
Endrin
Endosulfan sulfate
δ-BHC
Chlordane
Toxaphene
PCB-1242 (Arochlor 1242)
PCB-1254 (Arochlor 1254)
PCB-1221 (Arochlor 1221)
PCB-1232 (Arochlor 1232)
PCB-1248 (Arochlor 1248)
PCB-1260 (Arochlor 1260)
PCB-1016 (Arochlor 1016)

TABLE A-5. METALS AND OTHER COMPOUNDS

Metals, total	Others
Antimony	Asbestos
Arsenic	Cyanide
Beryllium	
Cadmium	
Chromium	
Copper	
Lead	
Mercury	
Nickel	
Selenium	
Silver	
Thallium	
Zinc	

GLOSSARY

acute toxicity: Toxic effects to an organism due to a short-term exposure.

Ames test: Microbial mutagenicity bioassay developed by Dr. Bruce N. Ames.

cytotoxicity: Toxicity to mammalian cells.

EC_{50}: Effective concentration at which 50% of the test organisms reach the desired effect. The "effect," for example, can be growth inhibition or stimulation.

in vitro: Describing a biological reaction which can be performed outside the living organism, such as in a test tube.

LC_{50}: Lethal concentration fifty - calculated concentration of substance which is expected to cause death in 50% of the test organism population, as determined from their exposure to the substance.

secondary effluent: Textile wastewater treated by areated lagoons and clarified.

steady state: A condition of operation of a technology in which operating parameters are constant with respect to time.

tertiary effluent: Textile wastewater treated by technologies following aeration and clarification.

toxic pollutants: The 129 specific chemical species identified by EPA as a result of the consent decree.

toxicity: detrimental effects to the life of the test species such as fathead minnows, *Daphnia*, aglae, and cell cultures or an increase in the number of bacteria colonies in the Ames test (since the Ames test is a back-mutation test) as a result of exposure to the effluent sample.

CONVERSION FACTORS AND METRIC PREFIXES (13)

CONVERSION FACTORS

To convert from	to	Multiply by
Grams/meter (g/m^3)	Milligrams/liter	1.0
Kilogram (kg)	Pound-mass (avoirdupois)	2.205
Meter^3 (m^3)	Gallon (U.S. liquid)	2.642×10^2
Meter^3 (m^3)	Liter	1.0×10^3

METRIC PREFIXES

Prefix	Symbol	Multiplication factor	Example
Kilo	k	10^3	5 kg = 5×10^3 grams
Milli	m	10^{-3}	5 mg = 5×10^{-3} gram
Micro	μ	10^{-6}	5 μg = 5×10^{-3} gram

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(13) Standard for Metric Practice. ANSI/ASTM Designation: E 380-76$^{\varepsilon}$, IEEE Std 268-1976, American Society for Testing and Materials, Philadelphia, Pennsylvania, February 1976. 37 pp.

TECHNICAL REPORT DATA

(Please read Instructions on the reverse before completing)

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16. ABSTRACT The report gives results of a study concerned with BATEA for the textile manufacturing industry. The level of removal of specific toxic pollutants and toxicit (measured by results of bioassays) attained by selected tertiary systems treating secondary effluents from textile plants was examined. Tertiary treatment systems (unit processes arranged in various ways) were ranked according to their apparent capabilities for removing specific toxic pollutants and toxicity. Unit processes used included flocculation/sedimentation, multimedia filtration with and without precoagulation, granular activated carbon adsorption, and ozonation. The assessment of the treatment systems was based on specific toxic pollutant analysis data and on bioassay data gathered at eight textile plant locations where the treatment systems were tested. Samples from secondary and tertiary effluent streams were analyzed for spe cific toxic pollutants. Based on apparent specific toxic pollutant removal and toxicit removal, multimedia filtration-activated carbon was the best system. Tertiary treatment systems whose effluents contained a high level of residual coagulant appeared to be more detrimental to water quality, based on bioassays, than systems not using coagulants.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution	Pollution Control	13B
Assessments	Stationary Sources	14B
Textile Industry		11E
Waste Water		
Toxicity		06T
Bioassay		06A

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